THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Influence of Irradiation on Metallic Materials

C. A. BRUCH, W. E. McHUGH and R. W. HOCKENBURY: 'Variations in Radiation Damage to Metals.'

Jnl. of Metals, 1956, vol. 8, Oct., Sect. II; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 1362–72. T.P. 4276E.

The report deals with exposure tests on six metals (copper, nickel, titanium, zirconium, iron, molybdenum), and on molybdenum-containing austenitic chromium–nickel steel, irradiated in approximately equal exposure conditions in the Materials Testing Reactor at the Knolls Atomic Power Laboratory, General Electric Company, Schenectady.

Detailed information is given on the composition and condition of the materials used in the experiments, the test conditions, and the properties of the materials before and after irradiation. The general effects of irradiation were to increase strength and electrical resistivity, and to lower ductility. It is pointed out that the theory of radiation damage to metals is at present incomplete, and there is a lack of knowledge concerning many important factors. The theoretical aspect is, however, discussed on the basis of some observations made to date.

On an empirical basis, several consistent trends were found in the damage observed on the materials tested in these experiments. It is indicated that two factors strongly influence the magnitude of the changes caused by irradiation: these are the crystal structure of the metal, and the pre-irradiation value of the property being studied. While it is recognized that these observations cannot, in view of the relatively limited data, be considered conclusive, it is believed that such trends may be a useful guide for future experimental work.

Vibration of Castings during Solidification

See abstract on p. 18.

Sodium-Hydride Descaling Process

See abstract on p. 21.

Welding of Dissimilar Materials

HENRY WIGGIN AND CO., LTD.: 'INCO-Rod 'A': A New Welding Electrode.'

Publn. No. 1134,* 1956; 8 pp.

The electrode described has been developed especially for use in joining dissimilar alloy combinations by the metal-arc process. Sound, high-grade welds can thus be produced in a large proportion of the cases involving the use of dissimilar metals such as stainless steels, iron, alloy and mild steels, and high-nickel alloys. A table indicates the properties obtainable in welds made with the Inco-Rod 'A' between a wide variety of ferrous and non-ferrous materials. Some general recommendations are made on operating technique.

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NICKEL

Creep of Nickel

J. WEERTMAN and P. SHAHINIAN: 'Creep of Polycrystalline Nickel.'

Jnl. of Metals, 1956, vol. 8, Oct., Sect. II; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 1223–6.

Minimum creep rates of nickel samples were measured in the stress region $2{\cdot}5 \times 10^7$ to $2{\cdot}8 \times 10^9$ dyne per sq. cm., in the temperature range 400° to 1100°C. The creep rate appears to be proportional to $(\text{stress})^{4\cdot6}$ at stresses below 7×10^8 dyne per sq. cm. The activation energy of creep is approximately 65,000 cal. per mol.

Low-Temperature Properties of Nickel and of Nickel Alloy Steels

E. T. WESSEL: 'Some Exploratory Observations on the Tensile Properties of Metals at Very Low Temperatures.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 3; 26 pp.

The tensile properties and flow characteristics of selected materials were determined over a range extending from −269°C. to relatively elevated temperatures. The choice of materials was dictated by the requirement that three different lattice structures should be represented:—

Low-alloy nickel–chromium and nickel-chromium-molybdenum steels (quenched and tempered)	Body-centred cubic
Beta brass (annealed)	
Commercially pure nickel (annealed)	Face-centred cubic
Zirconium (annealed)	Close-packed hexagonal

All these materials exhibited appreciable ductility at the very low temperatures used: plastic deformation occurred primarily by a series of repeated discontinuous yields. It was found that the temperature-dependence of the b.c.c. materials investigated did

not conform to any of the theoretical relationships yet proposed: the variation was particularly noticeable at the lower temperatures of test.

Heat Capacity of Nickel at Low Temperatures

J. A. RAYNE and W. R. G. KEMP: 'The Heat Capacities of Chromium and Nickel.'

Philosophical Magazine, 1956, 8th Ser., vol. 1, Oct., pp. 918–25.

Heat-capacity measurements were made, at liquid-helium temperature, on very pure chromium and nickel, using a specially adapted cryostat, which is described and illustrated. In both cases the measurements give γ values lower than those obtained by former observers. The values of Debye temperature also differ significantly from those of previous work; it was found that $\theta = 630°K.$ (357°C.) for chromium and $\theta = 440°K.$ (167°C.) for nickel, in contrast to the older figures of 418°K. (145°C.) and 413°K. (140°C.), respectively. These new results help to resolve the apparently anomalous nature of the θ *vs.* T curves for the two metals. The influence of the magnetic heat capacity, on the results for nickel, is also discussed.

Reactions of Molten Sodium Hydroxide with Nickel and Nickel-containing Alloys

D. D. WILLIAMS, J. A. GRAND and R. R. MILLER: 'The Reactions of Molten Sodium Hydroxide with Various Metals.'

Jnl. Amer. Chemical Soc., 1956, vol. 78, Oct. 20, pp. 5150–5.

This paper is one of many which have recently indicated the growing importance of the application of liquid metals as heat-transfer media.

Among the obvious requirements for such materials are thermal stability and inertness with respect to the container material. One of the lower-melting and more thermally stable materials of this type is sodium hydroxide, and this paper presents chemical-reaction data on metal/sodium-hydroxide systems, clarifying many of the observations made with regard to corrosion and mass transfer in such systems.

The information given includes results of tests made at temperatures up to 1000°C., establishing the nature of the reactions of the following metals and alloys with sodium hydroxide:—nickel of varying degrees of purity, copper, nickel-copper alloy (Monel), gold, iron, chromium, titanium, tantalum, beryllium, manganese, silver, cobalt, nickel-chromium steels, iron-nickel alloy (64–36 per cent.) and nickel-chromium-iron alloy (64–17–17 per cent.).

Influence of Irradiation on Metallic Materials

See abstract on p. 1.

Special Corrosion-Resisting Applications of Nickel

See abstract on p. 24.

Analysis of Non-Ferrous Metals: Review

See abstract on p. 6.

Estimation of Nickel in Plating Solutions

See abstract on p. 5.

Influence of Nickel on Plants

A. H. KNIGHT and W. M. CROOKE: 'Interaction Between Nickel and Calcium in Plants.'

Nature, 1956, vol. 178, July 28, p. 220.

Details are given of a split-root technique which was developed to study the effect of nickel on calcium absorption in plants.

ELECTRODEPOSITION AND OTHER COATING METHODS

Nickel Plating: Reference Data

MOND NICKEL CO., LTD.: 'Nickel Plating for Engineers.' Publn. 952*, 1956; 72 pp.

Progress during recent years has brought commercial nickel-plating technique to the stage at which it is possible, by varying the composition of the plating solutions and the conditions of operation, to produce nickel coatings of controlled and consistent quality, suitable for use in widely varying applications, in an expanding range of industries. It is the purpose of this publication to describe some of the solutions which are in common use to-day, and to discuss conditions for operating them, with particular reference to the mechanical properties of the deposits which can be obtained from them. It is believed that this information, together with a knowledge of the limitations of nickel plating (which are also discussed in detail), will contribute to a better understanding between designer, planner, production engineer and plater, and thus advance the efficient use of nickel plating, on a sound economic basis.

The scope of the brochure is outlined below:—

The Nature of the Nickel-Plating Process.

Surface Preparation of the Basis Metal.
(Steels and non-ferrous metals in general; zinc-base die castings, aluminium and its alloys, nickel-chromium-base alloys, and nickel-chromium steels.)

Various Nickel-Plating Procedures.
(Anodes; Watts-type, all-chloride and sulphate solutions; bright-nickel solutions of the nickel-cobalt and organic types; barrel plating; sulphamate solutions; electrodeposition of tin-nickel; deposition of nickel-phosphorus alloys and electroless plating; black-nickel solutions.)

Mechanical Properties of Nickel Deposits.
(Tensile properties, hardness, internal stress, fatigue, and effect of heat-treatment on properties.)

Plating Practice.
(Covering and throwing power of solutions; plating to specification, racking, stopping-off, etc. Rough deposits, gas pitting, rinsing and drag-over contamination, purification of solutions; stripping of deposits.)

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Applications.
(Decorative, corrosion-resisting and 'engineering' deposits; machining of deposits; electroforming; plating of non-conductors; wear-resisting applications.)

Plant.
(Tanks, filtration, heating equipment, agitation procedure, electrical supply, automatic plants.)

Testing of Deposits.
(Tests for determination of corrosion-resistance, porosity, thickness, adhesion, stress and ductility.)

The final section contains some reference tables giving information of general use to platers and others, e.g., thicknesses of plating electrodeposited in given times under specified conditions, physical constants of commercially pure electrodeposited metals, factors for use in calculating composition of plating solutions, and conversion factors (length, area, volume, etc.) for values likely to be encountered in plating specifications. The text of the publication is supported by a *bibliography* of some 30 references, selected from the wide literature of the subject. All sections are extensively illustrated.

Nickel Salts for Plating: Chromium Plating of Nickel Silver

DEUTSCHER NORMENAUSSCHUSS: 'Electroplated Coatings: Nickel Salts for Nickel-Plating Solutions.' D.I.N. Specification 50 970 (Revised Edn.).

Werkstoffe und Korrosion, 1956, vol. 7, Oct., pp. 606–8.

The original form of this specification, issued in March 1953, no longer corresponds to the current stage of development in nickel plating, particularly with regard to bright plating. Platers are calling for salts of higher purity than those required by the earlier specification, and it has therefore been decided by the German standardizing authority to provide for two grades of nickel sulphate, simultaneously to make certain changes in the requirements laid down for testing electrodeposited nickel coatings, and to provide for the use of the Hull cell in control of plating solutions before and during use. These modifications are incorporated in the tentative specification now issued.

The following are the main provisions:—

Nickel Content of Salts

Salt	Nickel + Cobalt % min.
Nickel Sulphate, Grade A	20·8
Nickel Sulphate, Grade B	20·6
Nickel Chloride	23·7

Purity Requirements for the Nickel Salts

Salt	Permissible Impurities							
	Co %	Zn %	Fe %	Cu %	Pb %	Cd %	As %	Constituents insoluble in n/10-Sulphuric Acid %
Nickel Sulphate, Grade A	2·00	0·010	0·005	0·002	0·002	0·005	0·001	0·05
Nickel Sulphate, Grade B	2·00	0·05	0·02	0·01	0·01	Not specified	Not specified	0·2
Nickel Chloride	2·00	0·05	0·05	0·01	0·02	0·02	Not specified	0·2

A plating solution produced with nickel sulphate of Grade A and containing 200 g./L. nickel sulphate shall have a pH value of not less than 3·5.

The nickel sulphate of Grade B shall not contain more than 0·1 per cent. free acid.

A further tentative specification, issued on the same date, covers the 'Electrodeposition of Chromium on Brass and Nickel Silver'. This specification, DIN 50 966, covers direct chromium plating of these materials, to thicknesses of 3, 6 or 9 μ. It is noted that there is not yet a sufficiently firm basis of knowledge for recommending procedure for direct deposition of thicker coatings. A warning is given that the corrosion-resistance of chromium-plated cold-worked brass is likely to be unsatisfactory.

Addition Agents in Nickel Plating

R. B. FISCHER and C. E. RING: 'Addition Agents in Nickel Plating.'

Plating, 1956, vol. 43, Nov., pp. 1338–41.

The programme of the research described was planned to investigate certain phases of the brightening mechanism of addition agents, by isolating, as far as possible, the effects of the addition agents from those of all other variables in the plating bath. The basis metal (copper), the surface preparation of this

base, the temperature, pH, current density, agitation and dimensions of the plating apparatus were all kept constant throughout the tests. The two addition agents, which were systematically varied, were 1,5-naphthalene disulphonic acid and saccharin. The deposits were examined by means of measurements of specular reflectivity and diffuse reflectivity, and by X-ray diffraction, electron diffraction and electron microscopy.

The Watts-type solution used was of the following composition:—

	g./L.
$NiSO_4.6H_2O$	330
$NiCl_2.6H_2O$	45
H_3BO_3	30

After precipitation and analysis, the pH was adjusted, by means of concentrated H_2SO_4, to 2·5, and re-adjustment was made as needed after each plating experiment. A current density of 50 amp./sq. ft. was used.

Thick (0·001 in.) and thin (2×10^{-6} in.) deposits were prepared from solutions containing 0–20 g. of 1,5-naphthalene disulphonic acid per litre, and two series of thick (0·001 in.) deposits were made from baths containing 0–10 g. of saccharin per litre.

The observations on the thin coatings showed that 1,5-naphthalene disulphonic acid had no effect on the physical characteristics of the deposits, indicating that the properties of a thin nickel electrodeposit are governed primarily by the nature of the surface of the basis metal. The physical properties of thick deposits were, however, considerably affected by both addition agents. Up to an optimum concentration each compound increased the specular reflectance but decreased the diffuse reflectance, made the surface of the deposit smoother, and lessened the surface grain size and crystal size. The optimum concentrations were about 0·3–0·5 g./L. for 1,5-naphthalene disulphonic acid and about 0·1–0·2 g./L. for saccharin. It is thus clear that the increase in brightness caused by the addition agent is accompanied by, if not actually caused by, increase in surface smoothness and decreasing crystalline or surface grain size.

Electrodeposition of Nickel on Iron Parts made by Powder Metallurgy

W. H. SAFRANEK and M. W. WIRTH: 'Selecting Electroplates for Iron Powder Parts.'

Materials and Methods, 1956, vol. 44, Nov., pp. 104–9.

This report summarizes the results of a comprehensive investigation carried out at the Battelle Memorial Institute, for Detroit Arsenal. The main conclusions drawn are as follows:—

'1. High-density and resin-impregnated parts have better corrosion-resistance after plating than low-density parts.

'2. Copper in resin-impregnated parts reduces corrosion-resistance.

'3. An acid copper-sulphate bath produces better copper deposits than a copper-cyanide solution.

'4. Nickel-sulphamate and zinc-sulphate baths produce, respectively, the best nickel and zinc plates.'

The factors which exert the most important effects on the corrosion-resistance of electroplated powder parts are the density and the copper content of the part and the throwing and covering power of the plating solution. In this report the compositions of solutions which may be used, and suitable operating conditions, are discussed in detail, with reference to the plating of iron powder parts with cadmium, zinc, nickel, chromium and copper.

The conditions recommended for electrodeposition of nickel are shown in Table I on page 5.

The various types of coating discussed are evaluated on the basis of appearance, corrosion-resistance and other characteristics. Some of the observations made with regard to nickel coatings are noted below:—

'*Nickel Plates.* A uniform and smooth nickel plate can be obtained, on low-density and impregnated parts, in both a Watts-type nickel-sulphate and a nickel-sulphamate solution. Pitting, does however, appear on high-density parts plated in a Watts-type bath, even when a wetting agent is used. This pitting is evidently caused by hydrogen bubbles, and cannot be eliminated by raising or lowering current density or by increasing agitation. In contrast, plates deposited on high-density powder parts in the sulphamate bath appear satisfactory, and show no traces of pitting. Apparently the sulphamate solution deposits nickel with better efficiency and with less hydrogen gassing.'

'On copper-bearing low-density powder parts plated with cadmium, zinc or copper discolouration has been observed after storage. No discolouration has been found, however, on chromium- or nickel-plated low-density parts, or on high-density impregnated parts plated with cadmium, zinc or copper.

'In salt-spray tests it was found that high- and low-density and impregnated parts plated with nickel, chromium or copper (sulphate bath) were about equal in resistance to corrosion. In general, plated iron-powder parts which were copper-free were found to be more resistant to salt spray than were copper-bearing parts.

'In humidity tests powder parts plated in a nickel-sulphamate bath showed less corrosion than any other plated parts. Corrosion products consisting of small pinpoints of rust were found on these specimens, after only 24 hours, but after 1,042 hours the spots had not appreciably increased, and they could easily be washed off. Most of the copper and chromium-plated parts also showed evidence of rusting after 24 hours, but on continued exposure they acquired a tenacious black film. Parts plated with zinc showed a considerable amount of white corrosion product after 24 hours' exposure: in general, cadmium-plated parts gave better behaviour.'

Table I

Bath Composition		Operating Conditions	
Watts-type Nickel-Sulphate—		Temperature	110°F. (43°C.)
Nickel sulphate—$NiSO_4 . 7H_2O$	240 g./L.	Current density	45 amp./sq. ft.
Nickel chloride—$NiCl_2 . 6H_2O$	45 „	Agitation	30 c.p.m., 2½-in. stroke
Boric acid	30 „	Anodes:	
Wetting-agent solution	1·0% by vol.	External	Bagged, cast-nickel ovals
pH	4·8	Internal	¼-in. dia. nickel rods
		Anode voltage	1·6 v.
		Time to deposit 0·5 mil.	20 min.
Nickel-Sulphamate—		Temperature	120°F. (50°C.)
Nickel sulphamate—$Ni(H_2NSO_3)_2$	450 g./L.	Current density	30 amp./sq. ft.
Boric acid	30 „	Agitation	40 c.p.m., 2-in. stroke
Anti-pitting agent	0·375 „	Anodes:	
pH	4·0	External	Bagged, cast-nickel ovals
		Internal	¼-in. dia. nickel rods
		Anode voltage	2·3
		Time to deposit 0·5 mil.	30 min.

Estimation of Nickel in Plating Solutions

F. BRAKO: 'Analysis of Nickel in Plating Baths.'
Metal Finishing, 1956, vol. 54, Sept., pp. 61–4.

In an aqueous medium, nickel ions combine with purple murexide dye (*ibid.*, 1954, vol. 52, p. 71) to form a highly-coloured lemon-yellow compound which is stable at a neutral or alkaline pH. The brilliant colour persists even in the presence of a large excess of ammonium hydroxide, to demonstrate a strong nickel-to-murexide bond. This reaction is utilized in the analytical procedure described in the present article. The murexide dye acts as indicator, and titration is with sodium-cyanide solution. The chemical mechanisms involved are discussed, recommended procedure is detailed, and results are given of typical analyses of various types of nickel-plating solution.

Determination of Zinc in Nickel-Plating Solutions

T. A. DOWNEY: 'A Volumetric Determination of Traces of Zinc in Nickel-Plating Solutions.'
Plating, 1956, vol. 43, Nov., pp. 1335–41.

The increasing number of zinc-base die castings now being nickel plated, and the stringent specifications governing bright-nickel coatings have emphasized the significance of the possible influence of zinc impurities in the plating baths.

A method for determination of zinc in such solutions was described by E. J. SERFASS *et al.* (*ibid.* 1949, vol. 36, p. 818), and this gives satisfactory results, but the procedure has the disadvantage of being somewhat lengthy and requiring the use of unstable reagents, which must be frequently prepared. It also necessitates the preparation of a calibration curve and the use of a colorimeter.

KINNUNEN and WENNERSTRAND (*Chemist Analyst*, 1953, vol. 42, June, pp. 30–5) described a volumetric method for the determination of zinc in nickel-sulphate salts, but did not extend the application of the method to the determination of zinc impurities in nickel-plating solutions, nor did they consider the influence of all the impurities which might be in the solution, on the accuracy of the results obtained. The present paper describes the application of the Kinnunen method and the effects of common impurities in nickel solution.

Basically, the procedure consists of two parts:—

The first is the extraction and separation of the zinc as a zinc-thiocyanate complex, into an amyl-alcohol/ether mixture. This separates the zinc from the nickel solution and from any impurities present. The second stage is the complexometric titration of the extracted zinc, with ethylenediaminetetracetic disodium salt.

The recommended procedure is relatively simple and rapid and does not call for the use of any expensive apparatus. The only impurity found to cause interference was cadmium.

Hardness of Electrodeposited Tin-Nickel Alloy Coatings

V. R. RAMANATHAN: 'Hardness of Electrodeposited Tin-Nickel Alloy on Brass.'
Trans. Inst. Metal Finishing, 1957, vol. 34, pp. 1-7; *Bull. Inst. Metal Finishing*, Autumn 1956.

Hardness determinations on tin-nickel alloy deposits of three thicknesses (0·002, 0·001 and 0·0005 in.) on brass indicate that the hardness value of the deposits is of the order of 710 D.P.N. and that provided that the thickness of the coating is sixteen times the depth of penetration of the indenter, the measured hardness is consistently characteristic of the deposit, and is uninfluenced by the nature of the base metal.

NON-FERROUS ALLOYS

Nickel-Chromium-Titanium-Aluminium Alloys: Constitution

See abstract on p. 10.

Nickel-Cobalt Alloys Showing High Damping Capacity

A. W. COCHARDT: 'High-Damping Ferromagnetic Alloys.'
Jnl. of Metals, 1956, vol. 8, Oct., Sect. II; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 1295-98. T.P. 4322E.

The purpose of the investigation described was to survey the magneto-mechanical damping of ferromagnetic alloys having Curie temperatures above about 600°C. Such materials would be suitable for use in steam-turbine blades operating at the higher temperatures now becoming usual: the material currently employed, a high-chromium steel, has properties which are adequate up to only about 540°C.

In view of the sparsity of data available on damping or on irreversible magnetostriction under stress, it was necessary to examine the properties of a very large number of alloys: data on 55 different compositions based on nickel, iron and cobalt are presented in this paper. Full details are given of the processing of the alloys, of the damping apparatus employed, and of the characteristics of the individual materials.

Consideration of the all-over properties of the alloys examined indicates a cobalt-nickel base as being specially suitable for the purpose. Binary alloys containing 20-50 per cent. of nickel showed very high damping, and the cobalt-nickel alloys have the further advantage of being ductile at room temperature and having relatively high Curie temperatures. Such alloys are face-centred-cubic at elevated temperatures, and are inherently stronger than the high-chromium steel now used for steam-turbine blading. By only slight modification of composition, the alloys can be made precipitation-hardenable.

Properties of High-Chromium Alloys

See abstract on p. 14.

Reactions of Molten Sodium Hydroxide with Nickel-containing Alloys

See abstract on p. 2.

Special Corrosion-Resisting Applications of Nickel Alloys

See abstract on p. 24.

Analysis of Non-Ferrous Metals: Review

G. W. C. MILNER: 'Non-Ferrous Metallurgical Analysis.'
Analyst, 1956, vol. 81, Nov., pp. 619-50.

An introductory discussion of outstanding general developments in this field is followed by a critical survey of the chemical and physico-chemical procedures now available for the estimation of major and minor amounts of constituents which are of importance in non-ferrous metallurgy.

The elements considered are aluminium, antimony, arsenic, beryllium, bismuth, boron, cadmium, chromium, cobalt, copper, gallium, indium, iron, lead, magnesium, manganese, molybdenum, nickel, niobium and tantalum, silicon, silver, thorium, tin, titanium, uranium, zinc and zirconium. The review is supported by reference to 207 items of original literature.

A verbatim extract of the section relating to estimation of nickel is given below:—

'*Nickel*

Direct absorptiometric methods are suitable for the determination of small alloying amounts of this element. They are based on the red-coloured complex formed with dimethylglyoxime in ammoniacal solution, and much information is available on the best conditions for the formation of a stable complex. Direct methods are recommended for the determination of nickel in aluminium alloys[1], magnesium alloys[2], uranium metal[3], beryllium salts[4], and pure metallic tin[5]. For the analysis of copper-base alloys, however, a less direct method is used, because of the need for removing the major constituent. Various methods for removing copper have been reported, including electrolysis and deposition on aluminium foil, but the author[6] found the most expedient method to consist in precipitating the bulk of the copper with potassium iodide, followed by the formation of the nickel-dimethylglyoxime complex directly in the filtrate. The correction for the absorbancy of the small amounts of unprecipitated copper is readily obtained by taking absorbancy readings after the decomposition of the nickel complex with hydrogen peroxide.

'Polarographic methods are more direct than the absorptiometric method for the analysis of certain types of copper-base alloys. For example, by using the nickel step from an alkaline cyanide base solution, it is possible to determine the nickel content of cupro-nickels and aluminium bronzes by conventional polarography[7]. With alloys containing zinc, how-

ever, it is necessary to separate this constituent before recording the nickel step. This difficulty does not occur with tin-bronze samples on using a 0·2 *M* ammonium chloride—*M* ammonium hydroxide base solution in conjunction with the square-wave polarograph[8]. Moreover, it is possible to determine the zinc content simultaneously with the nickel content. Tin is the only constituent to prove troublesome, because of its insolubility in the alkaline-base electrolyte, but this element is readily removed as its volatile bromide in the initial solution of the sample. The square-wave polarograph is also applicable to the direct determination of nickel in aluminium alloys by using the peak from a 0·03 *M* potassium cyanide—*M* potassium hydroxide-base solution containing sodium sulphite to suppress the air oxidation of the manganese cyanide complex to manganese dioxide[9]. This method is direct and is applicable to a few milligrams only of sample, but it possesses no important advantages over the absorptiometric method. The polarographic method is extremely useful for the determination of small amounts of nickel impurity in cobalt metal and salts[10]. The polarogram is recorded from a pyridine base solution, under which conditions the nickel step is well separated from the cobalt step.

'Volumetric methods are suitable for the determination of larger alloying amounts of this constituent and one outstanding method involves the addition of a slight excess of a standardized potassium cyanide solution to complex the nickel, followed by titration with silver nitrate back to the potassium iodide end-point[11]. Nickel can also be titrated with EDTA, but there is so much interference in this titration from other elements that it is usually only applicable after the separation of the nickel from other constituents[12]. Flaschka[13], for example, has used this titration after the separation of the nickel with dimethylglyoxime. The use of EDTA is more important, however, in such analytical problems as the determination of nickel in the presence of small amounts of cobalt[14]. In a suggested method for this determination, sufficient EDTA is added to complex the nickel and cobalt and leave a slight excess. This excess is titrated with magnesium sulphate back to the colour change of the Eriochrome black T indicator. Then 2 to 3 ml. of hydrogen peroxide are added to the solution, followed by 1 g. of potassium cyanide. Under these conditions the cobalt complex is quite stable, so that the cyanide ions cause the liberation of the amount of EDTA equivalent to the nickel only. After the solution has been allowed to stand for about 5 minutes, the amount of liberated EDTA is determined by titration with magnesium sulphate to the production of the original colour of the indicator.'

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Determination of Copper in Alloy Steels

See abstract on p. 10.

Welding of Dissimilar Materials

See abstract on p. 1.

Welding of Titanium to Stainless Steel

See abstract on p. 24.

NICKEL-IRON ALLOYS

Martensitic Transformation in the Nickel-Iron System

L. KAUFMAN and M. COHEN: 'The Martensitic Transformation in the Iron-Nickel System.'

Jnl. of Metals, 1956, vol. 8, Oct., Sect. II; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 1393–1401. T.P. 4284E.

The authors draw attention to the relatively large literature already published on solid-phase equilibria and transformations in the iron-nickel system. The purpose of the work described was to extend existing information on the martensitic reaction and to develop a general thermodynamic treatment which would adequately cover equilibrium and non-equilibrium phase transformations.

The temperature of the start of martensite formation on cooling (M_S) and that of the commencement of austenite formation on heating (A_S) were determined on binary alloys containing 9·5–33·2 atomic per cent. of nickel. These findings were incorporated in a thermodynamic treatment of the system in the following manner:—

Equations were set up for the free energies of the α and γ phases, treating them as regular solid solutions of iron and nickel, and, by considering these phases

as solid solutions, it is shown that T_o, the temperature at which $\Delta F^{\alpha' \rightarrow \gamma} = O$, lies approximately halfway between M_s and A_s. This relationship was corroborated by a study of the effect of plastic deformation on M_s and A_s.

Magnetic Characteristics of Iron-Nickel-Aluminium Alloys

U. H. ROESLER: 'Study of Ferrous Ternary Diagrams in Relation to Magnetic Interactions: Fe-Ni-Al System.'

Jnl. of Metals, 1956, vol. 8, Oct., Sect. II; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 1285–9. T.P. 4302E.

The author directs attention to results of a thermodynamic analysis of the γ loop in iron alloys, recently published by Zener (*Trans. Amer. Inst. Mining and Metallurgical Engineers*, 1955, vol. 203, pp. 619–30), and reports experiments in which such analysis was applied to the ternary phase diagram Fe-Ni-Al. As a result, two separate parameters were established, $\Delta T_{\text{magnetic}}$, representing the influence of alloying elements on the magnetic transformation range, and $\Delta T_{\text{martensite}}$, describing the effect of such elements on a hypothetical martensite temperature.

These parameters were plotted against the concentration ratio Ni : (Ni + Al). By extrapolation of the resulting curves it was found that, according to the Zener theory, aluminium should raise the temperature range in which the atomic spins in iron become uncoupled from one another, but it is pointed out that this finding appears contradictory in view of the fact that aluminium lowers the Curie temperature of iron. Some suggested explanations of this anomaly are discussed.

Nickel-Cobalt-Iron Alloys Showing High Damping Capacity

See abstract on p. 6.

CONSTRUCTIONAL STEELS

Influence of Bainite in Nickel-Chromium-Molybdenum Steel

R. F. HEHEMANN, V. LUHANOV and A. R. TROIANO: 'The Influence of Bainite on Mechanical Properties.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 16; 23 pp.

A study was made of the influence of bainite on the mechanical properties of a low-alloy steel to S.A.E. 4340 specification, heat-treated to high and ultra-high strength levels. It was found that, in general, at a given strength level the presence of high-temperature bainite lowers the yield strength, reduction-in-area and impact-energy values obtainable, and the damage is progressive as the bainite is increased. As the temperature at which the bainite is formed is gradually lowered the effect of this phase on mechanical properties becomes less severe, until, with bainite which is formed near the M_s temperature, the reduction-in-area and toughness of mixed structures are equal, or slightly superior, to those of quenched-and-tempered structures.

The detrimental effect, on ductility, of the relatively soft high-temperature bainite is associated with the hardness differential between bainite and tempered martensite. Similar loss in ductility, at the higher strength levels, was observed with mixtures of soft and hard martensites. With small amounts of high-temperature bainite (up to 10–15 per cent.) the loss in ductility is relatively slight, indicating that heat-treatment which involves formation of small amounts of high-temperature bainite, prior to quenching to produce martensite, is commercially feasible.

Influence of Silicon on Structure and Properties of Nickel-Alloy Steel

J. VAJDA, J. J. HAUSER and C. WELLS: 'Effect of Silicon on Transverse Properties and on Retained-Austenite Content of High-Strength Steels.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 24; 34 pp.

The tests reported were made with the purpose of:—

(*a*) determining the effect of increasing silicon (1 and 1·5 per cent.), on the transverse mechanical properties of a wrought low-alloy nickel-chromium-molybdenum steel treated to tensile levels of 150,000–300,000 p.s.i. (67–134 tons per sq. in.),

(*b*) assessing the amount of retained austenite present in the steels, and

(*c*) ascertaining the influence of such retained austenite on mechanical properties.

It is pointed out that data on transverse ductility should be of particular value in connexion with the use, for aircraft and other structures, of ultra-high-tensile steels in which increased silicon is a significant feature.

The analysis of the basis composition used is given as carbon 0·40, silicon 0·25, manganese 0·71, sulphur 0·009, phosphorus 0·008, nickel 1·81, chromium 0·71, molybdenum 0·26, per cent. The test samples comprised end-quenched 7 in. × 7 in. rounds, examined in the fully-quenched and slack-quenched conditions: the experimental procedure used was described in an earlier paper (*ibid.*, 1954, vol. 46, p. 1331).

Among the conclusions derived from the results of the tests are the following:

It was confirmed that silicon additions improve hardenability and increase resistance to softening on tempering. Secondary hardening caused by silicon was observed in the steels when tempered,

in both conditions, in the range 260°–427°C. The maximum temperature at which silicon increased resistance to softening during tempering was found to be higher for the fully-quenched than for the slack-quenched steels.

The yield ratio in the end-quenched rounds was lowered by silicon additions. Marked improvement, in both yield strength and yield ratio, which resulted from tempering for 17 hours at 260°C. appeared to be essentially independent of the transformation of retained austenite, and it is considered that the improvement may be associated with relief of stress during tempering. Use of that tempering temperature and time on the steel containing 1·5 per cent. of silicon improved both transverse reduction of area and V-notch Charpy ductility. Such treatment also improved transverse ductility and toughness in the slack-quenched steel containing 1 per cent. of silicon (but not in the fully-quenched steel of that composition). When tempered at 427°C., however, the presence of both 1 and 1·5 per cent. of silicon adversely affected both properties.

From the results reported, it is concluded that aircraft-quality A.I.S.I. 4340 steel to which 1·5 per cent. of silicon has been added can be tempered at a temperature as high as 260°C., to give, in fully-quenched forgings, a tensile strength of about 280,000 p.s.i. (125 tons per sq. in.), together with relatively high transverse ductility and transverse toughness. Since the effect of silicon was determined over a very wide range of cooling rates, it may reasonably be presumed that the findings are applicable to large and massive forgings, as well as to smaller forgings.

Retained-austenite content, measured in standard Jominy test bars and in the 7-in. rounds subjected to end-quenching, was increased by the addition of silicon, especially in the slack-quenched structures. The authors propose an explanation of this effect of silicon. Retained austenite appeared to exert no significant influence on the mechanical properties of the steel.

Tempering of Steels: Composition/Treatment Relationship

L. D. JAFFE and E. GORDON: 'Temperability of Steels.' *Trans. Amer. Soc. Metals*, 1956, vol. 49, *Preprint* 14; 14 pp.

The authors describe a method for calculating, from the composition of the steel, the temperature required to temper it to a given hardness after quenching to martensite. The proposals are based on statistical analysis of hardness measurements on some 5,000 specimens from laboratory heats. The method is compared with other procedures which have been proposed for evaluation of tempering characteristics.

The compositions of the steels used to establish the formulae were chosen to give series in which one element at a time was systematically varied. In general, the alloying elements were varied at nominal carbon contents of 0·30, 0·70 and 1·00 per cent., and with or without chromium and molybdenum contents of 0·25 per cent. each. When not intentionally varied, nominal contents of the other elements were held at the following levels: manganese 0·75, silicon 0·25, nickel 1·50 per cent. On this basis, the influence of the following percentage ranges of elements was studied:—

Carbon	0·13, 0·30, 0·50, 0·70, 1·00.
Manganese	0·25, 0·50, 0·75, 1·25, 1·75.
Silicon	0·25, 0·50, 1·00, 1·50, 2·00.
Nickel	0, 0·50, 1·50, 2·50, 4·00.
Chromium	0, 0·30, 0·75, 1·25, 2·00.
Molybdenum	0, 0·12, 0·25, 0·50, 0·75.

Rates of Heating of Nickel-Alloy Steel Ingots

P. M. COOK and J. D. STRINGER: 'Increased Heating Rates for Steel Ingots.' *Jnl. Iron and Steel Inst.*, 1956, vol. 184, Nov., pp. 309–15.

The heating of ingots and billets for hot working is a relatively costly operation which, if carried out more slowly than is necessary, can be uneconomic. The current high cost of fuel, the shortage of steel, and the need for maximum utilization of plant, emphasize the importance of reducing heating time to a minimum.

The main factor governing the rate at which ingots may be heated is the susceptibiltiy of the material to 'clinking', a term used to describe the occurrence of transverse fractures caused by thermal-expansion stresses set up by the temperature gradient from surface to centre. An as-cast ingot structure, due to its lower ductility, is more susceptible to clinking than one which has been broken down by hot working, e.g., billet stock, and susceptibility increases with carbon and alloy content.

Following the recent publication of reports indicating the possibility of reduction in heating times, the present authors undertook experiments to determine how quickly ingots of various sizes and qualities could be raised to hot-working temperatures without suffering structural damage. This paper describes the experimental work and gives details of some works trials and applications of rapid methods of heating.

The tests were made on carbon steel, 2½ per cent. nickel-chromium-molybdenum steel (B.S. En 26), and high-speed tool steel, and the results indicate that the times conventionally used for heating both low- and high-alloy steels prior to hot working are unnecessarily long. The quality of finished products made from more rapidly heated steels has so far met commercial requirements, and it is stated that, as a result of the work reported, several steelmakers have shortened their heating times, with beneficial results. The advantages of such modification is urged, on economic grounds.

Nitriding of Steels

See abstract on p. 21.

Influence of Inclusions on Fatigue Strength of Nickel-Alloy Steel

H. N. CUMMINGS, F. B. STULEN and W. C. SCHULTE: 'Relation of Inclusions to the Fatigue Properties of S.A.E. 4340 Steel.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 23; 36 pp.

The significant influence of inclusions, on the fatigue strength of high-tensile steels, is well recognized: the authors introduce their paper by a review of some of the most pertinent literature. They then present a statistical analysis based on tests on some 1,000 specimens taken from a single heat of low-alloy nickel – chromium – molybdenum steel of aircraft quality, to S.A.E. specification 4340. A mill analysis gives the composition of the steel tested as carbon 0·40, silicon 0·31, manganese 0·77, sulphur 0·018, phosphorus 0·009, nickel 1·76, chromium 0·85, molybdenum 0·25, per cent. In addition, tests were made on over 100 specimens from a single heat of high-purity vacuum-melted steel of the same type. Smooth specimens were used, and the tests were made on R. R. Moore rotating-beam machines, at two speeds. All specimens were cut from bar stock and were longitudinal. The steels were tested at three levels of hardness, corresponding to tensile strengths of 140,000, 190,000 and 260,000 p.s.i. (62·5, 85 and 116 tons per sq. in.).

The tests revealed certain definite trends in the relation of inclusions to fatigue strength. These relationships are discussed at length, and from them is deduced an explanation of the mechanism of failure. On the basis of this proposed explanation, various predictions are made with regard to the behaviour of steels under fatigue conditions.

Low-Temperature Properties of Nickel-Alloy Steels

See abstract on p. 1.

Determination of Copper in Alloy Steels

L. J. A. HAYWOOD and P. SUTCLIFFE: 'Determination of Copper in Steel.'

Analyst, 1956, vol. 81, Nov., pp. 651–5.

Methods in common use for the determination of copper in steels involve separation as sulphide, either by use of hydrogen sulphide or sodium thiosulphate: see, for example, British Standard methods for determination of copper in magnet alloys and in carbon and low-alloy steels.*

Gravimetric methods of analysis involve the use of a relatively high conversion factor from oxide to metal, with the associated objection of the extreme difficulty of decomposing the sulphide during ignition and converting it to oxide.

Reference is made also to procedures employing sodium diethyldithiocarbamate, which are suitable for an absorptiometric finish, but the authors propose a method which they consider to be both quicker and simpler, particularly for routine batch determinations.

*B.S. 1121 series.

In determining copper in steel by the recommended technique, the element is complexed with bis*cyclo*hexanone oxalyldihydrazone and the resulting blue colour is measured by means of the Spekker absorptiometer within the range 570–600 mμ. The procedure is very rapid, simple and direct; no extraction, filtration or other separation is necessary, and the accuracy of the results is at least equal to that attained by methods hitherto available. The new method is believed to be suitable also for use on various ferrous and certain non-ferrous alloys.

Welding of Dissimilar Materials

See abstract on p. 1.

HEAT- AND CORROSION-RESISTING MATERIALS

Nickel-Chromium-Titanium-Aluminium Alloys: Constitution

A. TAYLOR: 'Constitution of Nickel-Rich Quaternary Alloys of the Ni-Cr-Ti-Al System.'

Jnl. of Metals, 1956, vol. 8, Oct., Sect. II; *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 1356–62. T.P. 4326E.

Nickel-rich alloys hardened by means of small additions of titanium and aluminium form an important series of high-temperature materials, because of their resistance to oxidation, high-temperature strength and excellent creep-resistance. Outstanding examples of this type are found in the Nimonic series of alloys, the main features of which have recently been described by Betteridge and Franklin (see *Nickel Bulletin*, 1956, vol. 29, No. 12, p. 217).

In the titanium- and aluminium-containing nickel-chromium-base alloys it is possible, by suitable heat-treatment, to precipitate, from the randomly ordered nickel-rich γ face-centered-cubic primary solid solution, a phase based on the ordered face-centred-cubic γ' phase, Ni_3Al. By adjusting the ratio of aluminium to titanium, the precipitation of needles of the hexagonal intermetallic compound η-Ni_3Ti may be effected.

In the investigation described the main interest was focused on the inter-relationships among the γ, γ' and η phases. Reports on the constitutional diagrams of the three contiguous systems, nickel-chromium-aluminium, nickel-titanium-aluminium and nickel-chromium-titanium have already been published by the author and a colleague; references to that work are given in the *bibliography* to the present paper, and the main observations made with regard to these systems are recapitulated, as an introduction to the report of the experimental work on the more complex alloys. The quaternary system nickel-chromium-titanium-aluminium was

studied, up to a temperature of 1150°C., on alloys containing more than 50 at. per cent. of nickel. It is shown that the region contains six single-phase areas: the face-centred-cubic nickel-rich primary solid solution γ, the face-centred-cubic ordered phase γ' based on Ni_3Al, the hexagonal-close-packed intermetallic compound η-Ni_3Ti, and three body-centred-cubic phases β_1-NiAl, β_2-NiTi and β_3-Ni_2TiAl. As the temperature is raised, the volume of the quaternary tetrahedron occupied by the γ phase increases considerably, while that of γ' decreases. The tie-lines across $\gamma+\gamma'$, $\gamma+\eta$, and $\gamma'+\eta$ two-phase fields lie almost exactly in the plane Ni_3Cr-Ni_3Al-Ni_3Ti: these three positions effectively outline a pseudo-ternary system with the $\gamma+\gamma'+\eta$ triangle corresponding closely to a tie-plane. There is strong evidence that a four-phase field links the binary phases γ, γ' and η with the ternary phase β_3-Ni_2TiAl.

Effect of Molybdenum on Phases in High-Temperature Alloys

H. J. BEATTIE and F. L. VERSNYDER: 'The Influence of Molybdenum on the Phase Relationships of a High-Temperature Alloy.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 35; 17 pp.

The paper reports studies of the influence of molybdenum, up to about 11 per cent., on the structure of a complex nickel-chromium-cobalt-base alloy containing titanium and aluminium. Particular attention was directed to the influence of molybdenum on the carbide phases $M_{23}C_6$ and M_6C. The effect of molybdenum was investigated by correlation of the results of light and electron micrographic examination, and X-ray and electron-diffraction studies.

The microconstituents identified were Ti(C,N), $M_{23}C_6$, M_6C of two kinds, and sigma phase. Cellular precipitation was observed, and its apparent association with the comparatively slow rate of precipitation of the carbide $M_{23}C_6$ is discussed.

Conditions regulating the stability of the carbides found in these alloys are described, in particular the instability of the simple carbide TiC, as compared with that of the complex carbide M_6C.

Sigma Phase in 25-15 Chromium-Nickel Steel

L. PRYCE, H. HUGHES and K. W. ANDREWS: 'The Occurrence of Sigma Phase in a High-Chromium-Nickel Steel.'

Jnl. Iron and Steel Inst., 1956, vol. 184, Nov., pp. 289-301.

An earlier related paper described the mode of occurrence of sigma phase in 25-15 chromium-nickel steel, giving particular attention to the influence of carbon content (*ibid.*, 1952, vol. 171, pp. 49-58; abstract in *Nickel Bulletin*, 1952, vol. 25, No. 6, p. 160). In that paper the results obtained were correlated with the iron-nickel-chromium equilibrium diagram, due allowance being made for the effects of other elements which may be present in such alloys.

Work on the same type of steel has been extended, with special reference to determination of the influence of silicon on sigma formation, and also to amplify some other aspects of the subject, and to assess the influence of sigma on mechanical properties. The additional steels used comprised two groups of four, one series with low carbon content, 0·12-0·13 per cent., allowing for formation of some ferrite at solution temperatures, and the other with carbon at 0·22-0·24 per cent., ensuring solely austenite at such temperatures. Within each group silicon was progressively increased from ½ to 2½ per cent. Full details of composition and of heat-treatments are given.

Investigation of the steels, in various conditions, included microscopic examination, analysis of electrolytically separated specimens of carbide and sigma phases, and hardness and bend tests on samples in the solution-treated condition and after re-heating at 700° and at 900°C. for various periods. The results are shown in a comprehensive series of photomicrographs, in tables recording the nature of the structures and the proportions in which they were present in steels in various conditions, and in graphs giving results of the hardness and bend tests as a function of composition and heat-treatment.

These investigations confirm conclusions drawn in the earlier paper with reference to the mechanism and mode of formation of sigma phase in 25-15 chromium-nickel steels. It is demonstrated that increasing carbon tends to promote a wholly austenitic condition at solution-treatment temperatures, whilst increasing silicon favours a duplex structure. The observations made establish that sigma phase forms much more rapidly in steels which are partially ferritic. Formation from an austenitic matrix proceeds after precipitation of carbide, and, after long periods of heating, to some extent replaces that constituent. The results of the further work are again correlated with the iron-nickel-chromium equilibrium diagram and are discussed in relation to a formula suggested by GOW and HARDER for calculating the influence of individual elements on structural condition (see *Trans. Amer. Soc. Metals*, 1942, vol. 30, pp. 855-919). The authors propose an improved formula, by means of which the composition of steels of this type can be adjusted to give the fully austenitic condition at solution temperatures. For example, with chromium 25, nickel 15, manganese 1 and carbon 0·2, per cent., silicon should be less than 1·2 per cent. The use of the formula permits selection of compositions which will ensure freedom from sigma formation even after prolonged heating at 900°C. At lower temperatures considerably smaller chromium contents would be necessary for complete avoidance of sigma. An alternative would be to raise the nickel content, but this alone would not be so effective.

Hardness and bend tests were in accord with the constitution revealed by microscopical and X-ray

examination. Smaller angles of bend were associated with increase in silicon, but attention is directed to the fact that very low ductility is usually reached only after prolonged heating within the sigma-formation range. For example, in most cases heating for 100 hours at 700° or 900°C. results in no serious loss of ductility, although there is some falling off in steels containing relatively high silicon. It is therefore probably not essential to insist on a sigma-free condition for all kinds of service, since the effects of this constituent are dependent on the size and distribution of the particles, which, in their turn, depend on composition and on time and temperature of heating.

'G' Phase in High-Temperature Nickel-containing Alloys

H. J. BEATTIE and F. L. VERSNYDER: 'A New Complex Phase in a High-Temperature Alloy.'

Nature, 1956, vol. 178, July 28, pp. 208–9.

Note on a grain-boundary phase observed in an alloy of the following composition:—carbon 0·05, chromium 15, nickel 26, molybdenum 1·15, silicon 0·50, titanium 2·0, aluminium 0·17, vanadium 0·2, iron 53·5, per cent. (Allegheny-Ludlum Steel Corporation's A-286 alloy).

Specimens of the phase (which has been arbitrarily designated *G*) were electrolytically separated, and were examined by X-ray diffraction and by semi-quantitative chemical analysis. The predominant element was found to be nickel, with appreciable amounts of titanium and silicon, and some iron. The phase is more resistant to acid attack than is TiC, presumably due to the silicon content. Microscopical studies indicated a direct relation between the silicon content of the alloy and the amount of *G* phase present, which, together with the chemical results, suggests that silicon plays a fundamental part in its formation.

Structures in Precipitation-Hardening High-Temperature Alloys

W. C. HAGEL and H. J. BEATTIE: 'Ageing Reactions in Certain Super Alloys.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 40; 26 pp.

Changes in the structure and hardness resulting from solution treatments at 1800°–2200°F. (980°–1205°C.) and ageing at 1200°–1600°F. (650°– 870°C.), for times up to 1,000 hours, were studied in three high-temperature materials of the types shown in the table below.

The results are presented in a detailed series of tables and by means of electron micrographs showing typical structures.

Structures in Titanium-Stabilized Austenitic Steels

T. V. SIMPKINSON: 'Metallography of Titanium-Stabilized 18-8 Stainless Steel.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 29; 27 pp.

The primary object of this paper is to illustrate the features typical of the range of microstructures which, under various conditions of time and temperature, can be developed in 18–8-type chromium-nickel titanium-stabilized steels. The composition of the steel used is given as carbon 0·078, silicon 0·86, manganese 1·00, chromium 18·21, nickel 10·60, titanium 0·38, aluminium 0·07, nitrogen 0·016, per cent.

The main observations are summarized below.

'In the as-cast condition the steel contained a small amount of titanium-carbide precipitate and a moderate amount of chromium-carbide precipitate, the latter being concentrated at grain boundaries and at the periphery of ferrite islands. Since, in this condition, much of the carbon was still in solution, heavy precipitation of chromium carbide occurred when the casting was heated in the 'sensitizing' range. Some sigma phase was present, in some of the ferrite islands located at the grain boundaries, but no sigma was detected in ferrite which was within the austenite grains.

'A large proportion of the chromium carbide was dissolved by heating for one to two hours at 900°C., or by heating at a higher temperature for a shorter period. Treatment at 955°C., for half an hour, was required to dissolve all the sigma.

'During initial heating of the cast steel at 900°–955°C. there was heavy precipitation of titanium carbide. Even when the temperature of initial heating was much higher (up to about 1150°C.) there was some precipitation, although the amount decreased as the temperature of heating was raised. Re-heating, at a higher temperature, of specimens which had been initially heated at 900°–955°C. resulted in re-dissolving part of the titanium carbide precipitated at the lower temperatures: the higher the temperature of re-heating, the more completely was the carbide re-dissolved.

'The heat-treatment usually specified for testing the susceptibility of austenitic steels to sensitization, *viz.*, 1–2 hours at 650°–675°C., produced a small

Alloy	C %	Cr %	Ni %	Co %	Mo %	W %	Nb %	Mn %	Si %	P %	S %	Cu %	Fe %	N %
N-155	0·14	21·19	19·50	20·38	2·85	2·98	1·20	1·80	0·38	0·018	0·016	—	bal.	0·16
G-18B	0·42	13·07	13·53	9·93	1·80	2·91	2·77	0·78	0·76	0·018	0·012	0·14	bal.	0·034
15-15N	0·09	15·20	15·67	—	1·44	1·09	1·14	1·34	0·76	0·013	0·011	0·20	bal.	0·023

amount of sigma in this steel, but it is considered unlikely that this proportion of the phase would have any important influence on the corrosion-test results. Heating of the steel for 100 hours in the sigma-formation range of temperature produced a large amount of that phase.'

An appendix to the paper gives the composition of the etching solutions used for delineation of the respective structures.

Preferred Orientation and Transformations in Stainless Steel Wire

K. W. ANDREWS: 'An X-ray Examination of Preferred Orientation and the Transformation $\gamma \rightarrow \alpha$ in a Stainless-Steel Wire.'

Jnl. Iron and Steel. Inst., 1956, vol. 84, Nov., pp. 274–86.

The paper describes the industrial processes through which wire passes in course of production and reports the structures observed at each stage of working. Stainless-steel wire of two compositions was used:—

Steel	C %	Si %	Mn %	Ni %	Cr %
A	0·10	0·55	0·63	9·80	19·16
B	0·62	0·6	0·51	8·11	18·1

The nitrogen content of the steels is specifically discussed, as a factor in determining the behaviour of the steels in processing, see below.

The extensive report of observations made at the various stages of working leads to the following conclusions:—

Austenitic stainless-steel wire of the 18–8 chromium-nickel type develops a preferred orientation during the wire-drawing process, and under normal annealing conditions this orientation is not removed. The orientation is considerably sharper towards the centre of the wire than at the surface.

The predominant component in the texture observed is represented by a cubic diagonal, i.e., a <111> direction lying parallel to the axis of the wire. There is also a small proportion of grains with a cubic edge <100> direction parallel to the axis. Other X-ray effects found are explained by the existence of twinned crystals.

The cold deformation involved in wire-drawing causes transformation of some of the austenite to ferrite: the amount formed depends on the composition of the steel. The proportion of ferrite increases towards the centre of the wire, and this feature was the subject of a special investigation, as a result of which it is concluded that an explanation advanced earlier by the authors (*ibid.*, 1949, vol. 162, pp. 424–7), is probably correct. Nitrogen content, although it may have a pronounced effect at the immediate surface of the wire, is not a determining factor at greater depths.

The ferrite phase possesses a preferred orientation, which does not, however, correspond to the texture usually observed in a body-centred-cubic metal. An interpretation of the orientation of the ferrite phase is advanced on the basis of its dependence on the lattice of the austenite phase from which it is formed.

High-Temperature Nickel-base Alloys

'Past, Present, and Future of High-Temperature Nickel-base Alloys.'

Jnl. of Metals, 1956, vol. 8, Oct., Sect. I, pp. 1445–9.

The article is based on a review presented by F. L. VERSNYDER at a Session on 'Behaviour of Materials at Elevated Temperatures' held at the Massachusetts Institute of Technology in 1955.

Brief reference is made to nickel-base high-temperature alloys as originating in the Marsh patents of 1906, and the development is traced through the work of British, U.S. and Continental investigators who later studied the improvement of such alloys, the addition of other elements, and special forms of heat-treatment.

A table summarizes the nominal compositions of 26 materials typical of nickel-base alloys now used for high-temperature service, and some observations are made on the influence of composition on properties. Melting, casting and processing technique is then considered, with special reference to problems introduced by the presence of titanium.

With regard to processing, attention is called to the advantages of extrusion (as compared with forging or rolling), in obtaining increased yields of bar stock from these refractory alloys. Mechanical properties required in high-temperature alloys are then considered, and the inter-relation of properties and structure are discussed by reference to typical materials. Some comparison is made between the high-temperature properties of nickel-base and cobalt-base alloys.

Finally, a warning is given with regard to prevention of recrystallization of the surface of nickel-base alloys during the finishing of blades and other components, and the avoidance of intergranular oxidation, which may result in cracking. Both problems can be solved by electromachining in the final stages of processing, thus removing the cold-worked layer and any intergranular penetration which has resulted from the manufacturing process.

Properties of High-Chromium Alloys

D. J. MAYKUTH and R. I. JAFFEE: 'The Mechanical Properties of Swaged Iodide-base Chromium and Chromium Alloys.'
Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 38; 10 pp.

Two earlier papers by the same authors had reported, respectively, the mechanical properties of iodide chromium and of eight iodide-chromium-base alloys, tested, in all cases, in the form of thin strip. The results were briefly as follows:—

(1) Iodide-chromium strip 0·040 in. thick was reasonably ductile in bending, down to −25°C., but exhibited no tensile ductility at room temperature. After recrystallization the strip had neither bend nor tensile ductility at room temperature.

(2) Of eight binary alloys made with iodide chromium, the only compositions containing at least 50 per cent. chromium which showed good hot workability and tensile ductility were those containing 40–50 per cent. of nickel or iron. As quenched from elevated temperature, the chromium-iron alloys were single-phase solid solutions, and the chromium-nickel alloys contained small amounts of a second phase consisting of the chromium-rich solid solution dispersed in an α nickel matrix.

This third paper deals with the properties of iodide chromium and its alloys with (*a*) iron, and (*b*) nickel, in the form of swaged bar, and compares them with the properties of the strip material referred to above. Determinations were also made of dynamic modulus of elasticity over the temperature range which is of interest in gas turbines.

The chromium used was prepared by thermal decomposition of chromous iodide: the values given below are quoted as indicative of the purity of the swaged bar:—carbon 0·006, nitrogen 0·001, oxygen 0·005, sulphur 0·001, total metallics 0·001, per cent.

The main points of interest in the test results are summarized below:

The wrought chromium bar showed a yield strength of 52,500 p.s.i. (23·4 tons per sq. in.), an ultimate strength of 60,000 p.s.i. (26·75 tons per sq. in.), elongation of 44 per cent. and reduction in area 78 per cent. These figures indicate the highest degree of ductility yet obtained in chromium metal, and the superiority of the material in this respect is considered to be due to its high degree of purity and its fibrous structure. A recrystallized specimen failed with a brittle fracture before the elastic limit had been reached, indicating that the fibrous structure is essential for optimum tensile ductility. Dynamic measurements showed the modulus of elasticity of the unalloyed chromium to be about 42,000 p.s.i. (18·75 tons per sq. in.) at room temperature: on the modulus/temperature curve there was a noticeable inflection at about 43°C.

Swaged and recrystallized chromium-base alloys containing 50 per cent. of iron or nickel, tested as bar stock, had excellent ductility, considerably better than that reported for the same materials in strip form. At 40 per cent. of iron, tensile ductility was low, and for the 40 per cent. nickel alloy, elongation was nil.

Modulus of elasticity was measured at temperatures up to about 800°C. The modulus increases with chromium content up to 50–60 per cent., but by only a relatively small amount.

Effect of Hot Working on High-Temperature Properties of Nickel-Chromium-Cobalt-Iron Alloy

J. F. EWING and J. W. FREEMAN: 'Influence of Hot-Working Conditions on High-Temperature Properties of a Heat-Resistant Alloy.'
Nat. Advisory Committee for Aeronautics, Tech. Note 3727, Aug. 1956; 134 pp.

The investigation described comprised a study of the principles governing the influence of hot-working conditions, on the high-temperature properties of complex austenitic alloys which are dependent on solution treatment or hot/cold work, rather than on strong age-hardening reactions, for development of high-temperature strength. The alloy chosen as typical of such materials was of the following composition: carbon 0·15, chromium 20, nickel 20, cobalt 20, molybdenum 3, tungsten 2, niobium 1, per cent., balance iron.

Working was carried out at several constant temperatures, to determine the influence of the amount of reduction at a given temperature: specific reductions at given temperatures, over a range of falling temperatures, were used to assess the effect of working over the normal falling-temperature range. More limited experiments were made to establish the effects of schedules which might involve re-heating to temperatures below and in the re-solution range, with reductions at low temperatures, where extensive precipitation occurs. In addition, some specimens were subjected to typical final solution, solution-and-ageing, and solution- and hot/cold-working treatments, to study the influence of prior working on response to thermal treatment. The investigation formed part of research on the fundamental metallurgy of heat-resistant alloys of the types used in propulsion systems for aircraft.

Evaluation of the effects of rolling was on the basis of rupture tests at 1200° and 1500°F. (650° and 815°C.), of creep rates during the rupture tests, and of creep rates for stresses of 25,000 p.s.i. (11·15 tons per sq. in.) at 1200°F. (650°C.) and 8,000 p.s.i. (3·6 tons per sq. in.) at 1500°F. (815°C.). Hardness determinations, examination of microstructures, and measurements of lattice parameter were employed to obtain data which would explain the metallurgical factors responsible for the effects of the respective treatments on high-temperature mechanical properties.

The following general conclusions are drawn:—

Medium-to-low strengths will result from large reductions made at nearly constant high temperatures. Very high strengths at 1200°F. (650°C.) and relatively high strengths at 1500°F. (815°C.) are characteristic of materials which have undergone gradual reduction over a falling-temperature range. Repeated working with abnormally low re-heat temperatures is one cause of very low strengths.

Hardness does not correlate with strength, since, after larger than optimum reductions hardness can continue to increase while strength falls off. In the rupture tests, ductility at 1500°F. (815°C.) was very sensitive to the amount of reduction.

Very uniform response to heat-treatment was obtained, suggesting that where response is variable the effect may be due to unidentified differences in the material from heat to heat. Variations in strength in the hot-worked condition appear to be due to a combined strengthening and weakening effect of working, on the structure of the alloy: strengthening was probably due mainly to strain-hardening: recrystallization, when it occurred, had a weakening effect. The observations on the effect of recrystallization suggest that weakening occurring even when recrystallization does not occur is due either to structural changes resulting from rolling, which induce recrystallization at the higher temperatures, or to a recovery process similar in nature to recrystallization: there may possibly be formation of sub-structures in the grains.

Considerable precipitation occurred in the alloy during working at temperatures within the range 1600°-2000°F. (870°-1095°C.), the effect being particularly noticeable at 1800°F. (980°C.). Such precipitation appeared to be detrimental to long-time rupture strength at 1200°F. (650°C.), but to have little effect at 1500°F. (815°C.), because of extensive further precipitation which took place during testing at the higher temperature. Temperature of working had a substantial effect on the properties at 1200°F. (650°C.), and also exerted a considerable influence on ductility in the rupture test at 1500°F. (815°C.).

The authors draw attention to a number of striking relationships between conditions of hot working and the properties obtained at high temperatures. When working at constant temperature, maximum rupture strengths at 1200°F. (650°C.) were obtained in material which had undergone a 15 per cent. reduction. Maximum rupture strengths at 1500°F. (815°C.) were obtained on material subjected to isothermal working at temperatures within the range 1600°-2200°F. (870°-1205°C.), but the optimum reductions were not constant. Maximum creep-resistance was in most cases associated with smaller reductions than those which gave maximum rupture strength.

Lattice parameters varied considerably with conditions of working and with cooling rate, but the reasons for such variation of structure are not yet understood. Grain-size did not in itself appear to be a controlling factor.

Titanium and Boron Additions to Stainless Steels

J. SALVAGGI and L. A. YERKOVICH: 'High-Temperature Rupture-Strength Properties of Chromium-Nickel Stainless Steels containing Titanium and Boron.'

Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 33; 19 pp.

In the early stages of gas-turbine and jet-propulsion development the need for rapid progress and the insistent calls for increasing efficiency necessitated the use of very highly alloyed materials, in order to provide ample margin of performance for the high-temperature service conditions involved. Design and operation of such units are now, however, becoming sufficiently stabilized to warrant closer consideration of the precise properties called for in varying conditions of operation, and a less empirical approach to the means by which the required characteristics can be obtained. There is, at present, a general trend towards examination of the potential usefulness, for some components, of materials of somewhat lower alloy content, and this paper reports one of the investigations made with this aim. It deals with the high-temperature strength which can be obtained in 18-8 chromium-nickel and 17-12 chromium-nickel-molybdenum steels (of the types containing carbon 0·07-0·3 per cent.), when modified by additions of titanium and/or boron.

The main emphasis was placed on survey of the high-temperature properties of a reasonably large number of compositions, without detailed consideration of fabrication and production aspects and other metallurgical characteristics. The materials were used in the form of 0·050-in. sheet, subjected to stress-rupture tests at 1500°F. (815°C.). Some tests were made also at 1350°F. (732°C.). The metallography of the titanium- and boron-modified steels was examined on specimens of the molybdenum-containing 17-12 type.

The results of the tests indicate that titanium, boron and carbon are all capable of improving the 100-hour rupture strength at 815°C., in both steels solution-treated in the range 2100°-2300°F. (1150°-1260°C.). Ductility at rupture was satisfactory, being in most cases better than that of the corresponding basis steels solution-treated at similar temperatures. In general, maximum strength at high temperatures increased with increase in carbon content. Within the range studied, rupture ductility was relatively little affected by variation in carbon, especially in the steels containing both titanium and boron. After given treatments, and at similar titanium, boron and carbon levels, the modified molybdenum-containing steels were found to have better high-temperature properties than the modified 18-8 chromium-nickel types.

In the steels containing both titanium and boron, maximum 100-hour rupture-stress values occurred at progressively lower titanium contents as the boron content was increased. High rupture strengths in the modified steels were favoured by the use of the higher temperatures for solution treatment, but this gain was associated with some drop in ductility at rupture. The latter effect was less pronounced in boron-containing steels than in those which contained only titanium.

Influence of Irradiation on Metallic Materials

See abstract on p. 1.

Effect of Environment on Creep Strength

P. SHAHINIAN: 'Effect of Environment on Creep-Rupture Properties of Some Commercial Alloys.' *Trans. Amer. Soc. Metals*, 1956, vol. 49, *Preprint* 34; 22 pp.

Creep-rupture tests were made, in air, in oxygen, nitrogen and hydrogen, and in vacuo, on materials of the types shown below.

The primary object of the research was to determine the influence of environment on creep behaviour at or near the usual service temperatures of the respective materials; the influence of environment in the presence of stress concentration was also investigated, by means of notched specimens of all the materials except the 12 per cent. chromium-vanadium steel and N-155 alloy. Each material was studied, for periods up to 500 hours, at a single temperature, ranging from 1100°F. (593°C.) for the chromium-vanadium steel to 1500°F. (815°C.) for the 'super alloys'. A gas-tight chamber used for the controlled-atmosphere tests is described and illustrated.

The following observations are recorded:

The rupture life and minimum creep rate of all the materials was affected by environment, and certain effects were common to all of them. Rupture life was longer in air than in oxygen, nitrogen, helium or vacuum. In general, longer lives were obtained in oxidizing than in inert atmospheres. Minimum creep rate was usually lowest in air, but no consistent effect was produced by the other atmospheres.

In alloys of relatively low ductility, S-816 and Inconel X, the presence of a notch greatly increased the sensitivity of the materials to environment. Inconel X at 1500°F. (815°C.) and 25,000 p.s.i. (11·15 tons per sq. in.) was notch-strengthened in air and oxygen atmospheres, but notch-weakened in nitrogen and in vacuum.

On the basis of suggestions made in published literature, in association with the authors' own observations, the following comments are made on the factors which may be significant with regard to the influence of atmosphere on properties:—

'Surface films may inhibit the generation and/or migration of dislocations.

'Internal oxidation may strengthen the material.

'An oxide film may mechanically strengthen the bulk material.

'Oxygen penetration into grain boundaries may occur, and result in improved creep-resistance.

'Oxidation may dull the crack tip, which would tend to retard propagation of cracks.'

Oxidation Characteristics of Nickel-Chromium Alloys

G. E. ZIMA: 'Some High-Temperature Oxidation Characteristics of Nickel with Chromium Additions.' *Trans. Amer. Soc. Metals*, 1956, vol. 49, *Preprint* 37; 26 pp.

Most of the investigations on the oxidation of nickel-chromium alloys have been concerned with materials containing 20 per cent. or more of chromium, and particular interest has centred on the 80-20 type, which has for many years been outstanding as the basis of various heat-resisting alloys.

In contrast with the influence of chromium when present in substantial amounts, it has been found that the addition of much smaller quantities (e.g., 2-10 per cent.) to nickel results in increased rate of oxidation. In the present paper an attempt is made to delimit more closely the harmful/beneficial composition boundary, and to relate the oxidation characteristics of alloys of varying composition to the nature of the oxides formed on them. The range covered was 0-17 at. per cent. chromium. Since the oxidation of nickel-chromium alloys within the range of composition studied is believed to be governed by NiO with chromium substitution, the paper is introduced by a discussion of the structure of NiO, with appropriate reference to the relevant literature.

In the experimental work reported the specimens were oxidized at 1096°C. (in aviators' breathing oxygen, purity 99·5 per cent.) for various periods, and the discontinuous-weighing method was employed, using an Oertling microbalance. Precautions were taken to prevent grain-boundary diffusion, and, in view of the severe quench-cracking of most of the oxides, each specimen was oxidized within its own quartz capsule.

As a basis of comparison, oxidation rates were determined for high-purity nickel, at temperatures of 980°, 1096° and 1260°C., under an atmosphere

Material	C %	Si %	Mn %	Ni %	Cr %	Mo %	Co %	Nb %	W %	Fe %	Other Elements %
D.M. 45	0·44	0·61	0·60	—	1·27	0·65	—	—	—	bal.	—
12% Cr-V Steel ..	0·15	0·19	0·48	—	11·5	—	—	—	—	bal.	V 1·05
A.I.S.I. 304 Steel ..	0·08	1·0	2·0	9	19	—	—	—	—	bal.	—
S-816	0·38	0·7	1·5	20	20	4·0	43	4·0	4·0	bal.	—
N-155	0·10	0·21	1·52	19·2	20·8	2·4	20·9	0·91	2·2	bal.	N_2 0·06
Inconel X	0·04	0·3	0·5	73	15	—	—	1·0	—	9	Ti 2·5 Al 0·7

of oxygen. The data, on both nickel and the alloys, are usefully correlated with the findings of other investigators.

It was found that increasing amounts of chromium n the NiO phase govern the rate of oxidation for alloys containing less than 2 at. per cent. of chromium. For alloys of higher chromium content a complex exchange among three phases, NiO, Cr_2O_3 and the spinel $NiCr_2O_4$, was observed. This chromium exchange was reflected in the lattice parameter of the NiO phase, and in the rate at which the rate of oxidation increased with addition of chromium. The author's work indicates that in the oxides of higher chromium content an interstitial diffusion mechanism may become dominant. Within the scope of composition covered by this work, the high oxidation stability of nickel-chromium alloys was confirmed as being associated with the presence of Cr_2O_3 in the oxide layer. The data on nickel, combined with those reported by Gulbransen and Andrew for lower temperatures, yielded an activation energy of 45·1 Kcal./mole for the oxidation of nickel.

Oxide Scales on Austenitic Nickel-Chromium Steels

H. J. YEARIAN, H. E. BOREN and R. E. WARR: 'Structure of Oxide Scales on Nickel-Chromium Steels.' *Corrosion*, 1956, vol. 12, Nov., pp. 561t-8t.

An earlier paper had reported the results of X-ray diffraction investigation of the scales formed on straight-chromium steels under various conditions. From the observations made, some relation between type of oxide and corrosion protection was developed, and the mechanisms of oxidation associated with the various conditions were discussed. The present paper reports similar investigations on nickel-chromium stainless steels and some higher-nickel nickel-chromium-iron and nickel-chromium alloys. The experimental conditions, selected to be identical with those used by Brasunas *et al.* in an extensive study of the behaviour of cast materials (*Proc. Amer. Soc. Testing Materials*, 1946, vol. 46, p. 129), comprised 100 hours' exposure at 1600°, 1800°, 2000° and 2200°F. (870°, 980°, 1093° and 1205°C.) in air which had been saturated with water at 90°F. (32°C.), flowing at a rate of 200 cm^3/min. Specimens about 1 in. × $\frac{3}{4}$ in. × $\frac{1}{8}$ in. were polished through 3/0 emery papers, vapour-degreased, and oxidized in a platinum-wound resistance furnace.

The materials exposed ranged from straight 18-8-type chromium-nickel steels to 80-20 nickel-chromium alloy, and were chosen to cover the maximum range of protective properties, using a minimum number of samples which would be representative of alloys in common use. Two steels of relatively high nickel-chromium content were used at two silicon levels, to evaluate the influence of that element.

X-ray diffraction examination showed that the scales formed on the materials tested were of two distinct types. The principal, and most significant constituent of the more protective type is Cr_2O_3, but a solid solution of Fe_2O_3-Cr_2O_3 may also be present, and in such cases the Fe_2O_3 content tends to increase with the increase in rate of attack at any given temperature. At very low rates of attack considerable amounts of $MnCr_2O_4$ occur when the alloy contains a few tenths per cent. of manganese, but as the rate of attack increases this manganese-chromite spinel is replaced by a nickel-chromium-iron spinel, the amount and the alloying content of which increase with rise in temperature and with increase in the nickel content of the alloy. On an 80-20 nickel-chromium alloy NiO is also present.

When the rate of attack becomes excessive the scale changes to a type which contains no Cr_2O_3, but consists of one or more Fe_2O_3-Cr_2O_3 solutions and the nickel-chromium-iron spinels. The ferric-chromic oxide solution tends to concentrate in the outer layer of the scale, and at that position contains less than 10 mole per cent. Cr_2O_3, but the proportion may increase in the inner layers of scale. Spinels are present in all the layers, but the alloying elements present in them increase in an inward direction.

The observations made are compared with published data and are interpreted on the basis of a theory of selective oxidation and depletion.

Specific Resistance/Structure Relationships in Nickel-Chromium Alloys

T. A. DUNTON and H. LEWIS: 'Heating-Element Materials: Relationship between Specific Resistance and Temperature.' *Electrical Rev.*, 1956, vol. 159, Oct. 5, pp. 599-601.

This article discusses the order-disorder reaction which occurs in nickel-chromium and nickel-chromium-iron alloys, as affecting their specific electrical resistance. The information given is based on tests made on two typical alloys, Brightray C (nickel-chromium) and Brightray B (nickel-chromium-iron).

The specific resistance of the alloys in the ordered condition is slightly higher than that of the disordered alloys, and the resistance at room temperature depends upon the degree of ordering achieved, which, in turn, is controlled by the rate at which the alloys have been cooled after annealing treatment. Specific-resistance/temperature relationships have therefore been determined for both slowly and rapidly cooled specimens: the results are graphically illustrated and the influence of composition, on the extent of the structural changes, is discussed.

These alloys are, however, normally supplied in one of two annealed conditions: 'bright-annealed' (as produced by treatment in an atmosphere which prevents oxidation), or 'oxy-annealed' (i.e., annealed in air, to produce an oxidized finish). Since the rate of cooling after these normal annealing treatments does not correspond to either of the extremes used in the experiments referred to above, further tests were made, on wires of various sizes, obtained from normal production materials in both conditions.

The conclusions to be drawn from both series of tests are that for precision applications of nickel-chromium and nickel-chromium-iron alloys, and for applications involving their use as highly accurate resistance materials at temperatures below 500°C. the effects of the order-disorder reaction, on specific

resistance, must be taken into consideration. It is emphasized, however, that the sensitivity of specific resistance to cooling rate need occasion no anxiety on the part of the manufacturers of heating appliances. The variations which can result from this cause are too small to affect efficient working in most designs of heating elements, and, in the majority of cases operating temperatures are above the range in which variations of resistance at a given temperature can occur.

Vibration of High-Alloy Castings during Solidification

J. JAGACIAK and J. W. JONES: 'Effect of Vibrations during Solidification on the Mechanical Properties of Castings of Gas-Turbine Materials.'
Foundry Trade Jnl., 1956, vol. 101, Nov. 22, pp. 595–603.

A report by HINCHLIFF and JONES (*ibid.*, 1955, vol. 99, p. 251) on vibration, at sub-sonic frequencies, of wedge-type castings of H.R. Crown Max steel during solidification showed that whilst the mechanical properties were primarily dependent on casting temperature, some improvement was effected by vibrational treatment. Complementary work is now reported, using H.R. Crown Max and Nimonic C (for compositions, see table below).

The aim of the work was to determine the potential value of vibrational treatment in production of cast gas-turbine blades, the assumption being that the non-uniformity of the as-cast structure would thus be overcome, giving a higher degree of mechanical strength than could be obtained in blades produced by conventional procedure.

The results of the second series of experiments, now reported, show that vibration in the sub-sonic range effects marked improvement in the properties of both materials. With H.R. Crown Max the greatest benefit is obtained at about 4,000–5,000 v.p.m.; for Nimonic C 75–85 v.p.s. gave better results. Typical data are shown in the table in the right hand column.

These improvements in mechanical properties were confirmed by changes in structure resulting from vibration; these were common to both materials. Large dendrites disappeared, giving way to a finer structure, approaching that characteristic of the annealed condition. Increase in density was also associated with the higher mechanical properties.

Further work will be necessary to determine whether all parts of a casting of varying section would benefit equally by vibrational treatment, but the results on the H.R. Crown Max casting, which varied in thickness from ½ in. to very thin section, appear promising.

The fact that two alloys of different compositions responded in a similarly favourable manner to vibrational treatment lends support to the theories advanced in the earlier paper with regard to the potential value of such technique.

It is pointed out that the electro-mechanical vibrator required is not an expensive item of equipment and that it is sufficiently robust to be used in the foundry.

Percentage Improvements obtained in Properties by Vibrational Treatment

	H.R. Crown Max.	Nimonic C
Ultimate Tensile Strength	14	11
Impact Strength ..	62	61
Elongation, %	30	25
Reduction of Area, % ..	40	28
0·1% Proof Stress ..	10	13

Cast Nickel Alloys: Production and Properties

C. M. HAMMOND and R. A. FLINN: 'Effect of Melting and Casting Atmospheres on Cast Nickel Alloys.'
Jnl. of Metals, 1956, vol. 8, Oct., Sect. I, pp. 1450–6.

The work described was designed to evaluate the properties obtainable in nickel-base casting alloys, and to determine the most favourable production and processing conditions. Survey of the literature indicated three types of established alloys as being specially promising: the nominal compositions and some properties are shown in Tables I and II, on page 19.

The following details of production are given:—

A good commercial investment practice was adopted as standard: wax patterns and an ethyl-silicate-base investment were used. Details are given of method of mould preparation.

The experimental alloys were melted and poured under three atmospheric conditions: air, argon-protected, and vacuum+argon. In most cases the melts were made from virgin metals.

The materials produced were evaluated on the basis of tensile properties and hardness at ambient temperature and of stress-rupture tests and measurements of elongation and reduction of area at elevated temperatures. Structure was correlated with composition and properties.

Material	C %	Si %	Mn %	S %	Ni %	Cr %	Al %	Ti %	Cu %	Fe %	W %
H.R. Crown Max. ..	0·20	1·60	0·40	—	11·50	23·00	—	—	—	—	3·00
Nimonic C	0·08-0·15	0·30-0·80	1·00 max.	0·015 max.	Bal.	19-21	0·40 max.	0·25	0·50 max.	2·00 max.	—

Table I

Alloy Type	C %	Cr %	Mo %	Al %	Fe %	Ti %	B %	Co %	Nb %
Guy ..	0·10	13	6·0	6·0	4·5	—	0·50	—	2·0
Inco 700 ..	0·10	15	3·0	3·0	·5	2·0	—	28	—
GMR 235 ..	0·15	15·5	5·25	3·0	10·0	2·0	0·075	—	—

Table III

Alloy Type	Published Properties	This Investigation		
		Air	Argon-Protected	Vacuum + Argon
Guy Alloy				
100-hour rupture strength				
p.s.i.	49,000	42,000	41,000	56,000
t.s.i.	21·9	18·75	18·3	25·0
% Elongation, 100 hour ..	2-5	3	1-3	7-10
Inco 700				
100-hour rupture strength				
p.s.i.	43,000*	37,000	43,000	42,000
t.s.i.	19·2	16·5	19·2	18·75
% Elongation, 100 hour ..	10*	2-4	3-4	21
GMR 235				
100-hour rupture strength				
p.s.i.	37,000–40,000	41,000	41,000	42,000
t.s.i.	16·5–17·85	18·3	18·3	18·75
% Elongation, 100 hour ..	6-10	6-9	3-7	14-19

* Solution treated and aged.

Table II

Alloy	100-Hour Rupture Strength		% Elongation at 100 Hours
	p.s.i.	t.s.i.	
Guy	48,000-50,000	21·5-22·3	3-5
Inco 700*	41,000-43,000	18·3-19·2	10
GMR 235	38,000-40,000	17·0-17·8	6-10

* Properties from wrought specimens, solution treated at 2160°F. (1180°C.) for 2 hours and air-cooled; aged at 1600°F. (870°C.) for 4 hours and air-cooled.

A summary of the results is given in Table III.

Among the points to which particular attention is drawn are the following:—

In all three types of alloy, melting under vacuum and casting under argon atmospheres improved two- to ten-fold the ductility under stress-rupture conditions at 1500°F. (815°C.). The air-melted and argon-protected heats contained 5–10 times as much nitrogen as was found in the vacuum and argon heats.

Martensitic Transformation during Machining of Austenitic Steels

E. F. ERBIN, E. R. MARSHALL and W. A. BACKOFEN: 'Martensitic Transformation in the Machining of Austenitic Stainless Steel.'
Trans. Amer. Soc. Metals, 1956, vol. 49, *Preprint* 27; 21 pp.

Numerous studies of austenitic stainless steel have

been concerned with the development of low-carbon martensite or ferrite during plastic working processes and under various conditions of testing, but transformations which may occur during machining have received less attention, although it is probable that some of the difficulties encountered in the machine shop are attributable to the strength-producing transformation.

In the work reported in this paper, planing and grinding tests were made under standardized conditions, at normal and sub-zero temperatures, the lower temperatures being selected because exposure in that range promotes the stress-induced transformation.

Two types of austenitic steel were used: see table below:—

Type	C %	Ni %	Cr %	Mn %	Si %
301	0·08	5·80	17·96	1·89	1·00
304	0·053	10·31	18·70	1·25	0·39

Type 301 was chosen as representative of steels which transform readily when plastically deformed at (and somewhat above) room temperature: Type 304 is a steel which transforms appreciably only at lower temperatures. Before testing, all specimens (taken from hot-forged and -rolled material) were annealed in dry hydrogen at 1090°C., and air-cooled. Measurements of martensite content before heat-treatment and before the cutting tests showed 18 per cent. by volume in Type 301 and 6 per cent. in Type 304.

In planing four tools were used, having, respectively, rake angles 15°, 25°, 35° and 45°: in all cases the clearance angle was 5°. The variation in rake angle was introduced in order to produce chips having different shear strains: the larger the rake angle the more knife-like the tool and the lower the shear strain in the chip. Grinding was done by means of a Doall surface grinder, using a silicon-carbide wheel $6\frac{1}{2}$ in. in diameter and $\frac{1}{2}$ in. wide. Full details are given of experimental set-up and conditions.

An X-ray diffraction technique was employed for quantitative determination of the extent of transformation at the machined surface and, in some cases, as a function of depth below the surface.

The results indicate that in planing the depth to which the strain-induced transformation penetrates is only a fraction of that of the cut. The maximum amount of transformation occurred at the surface, where, for all the shear strains produced in chip formation, it was greater in Type 301 than in Type 304, and increased as the temperature was lowered from normal to −78°C. For both steels, and at all temperatures, shear stresses increased with shear strain, whilst the quantity of martensite at the planed surface decreased. This finding is attributed to the increasing strain rate and greater rise in temperature accompanying chip formation at higher shear strains. Both shear stress and energy of cutting corresponding to any degree of shear strain were found to increase as the martensite content at the planed surface increased.

In grinding, the zone transformed extended to a much greater proportionate depth: in Type 301, at 20°C. and at −78°C., it was found to penetrate to about 0·003 in. (i.e., six times the depth of the cut of 0·0005 in.). In grinding, maximum transformation occurred underneath, rather than at the surface, a fact which is explained as being the result of simultaneous effects of strain and temperature, both of which are greater in grinding than in planing.

Tests on Type 301, at −78°C. and at −196°C., showed that the amount of martensite at the surface was even less than that found in the interior well below the zone affected by grinding, an observation which is explained as being due to the very high surface temperatures involved. Grinding energy increased rapidly with fall in temperature, more rapidly in the case of the 301 than in the 304 steel.

Dimpling of Stainless Steel for Insulating Blankets

J. E. HAGINS: 'Dimpling Stainless Steel Foil.'
Machinery (Lond.), 1956, vol. 89, Nov. 16, pp. 1126–7.

The article contains an illustrated description of operations involved in the preparation of stainless-steel foil used to cover the surfaces of absorbent insulating blankets which cover the high-temperature heat ducts in jet engines. The very thin foil used (0·002–0·004 in. thick) is fragile, and it was found that the handling of large flat sheets of such material resulted in bends or stretches which developed into cracks. When the foil was shaped to fit round parts it tended to wrinkle, and sometimes cracked.

The problem has been overcome by dimpling the sheet, in a unidirectional staggered pattern. The dimples ensure sufficient metal for stretching at critical radii, and the staggering of dimple marks prevents the formation of stress lines. Embossing in this manner has reduced shroud failures to a minimum, and it has been found that the flexible sheet so produced can be used to insulate a large variety of aircraft components. A further incidental advantage which has resulted from the use of the embossed material is that the dimples have increased the heat-radiation capacity of the foil by at least 100 per cent., with the result that the product is more efficient in service.

The method of assembly of the insulating blanket composites is described and illustrated in this article.

Cold Heading of Austenitic Steels

S. E. TYSON: 'Cold-Headed Parts from Austenitic Stainless Steels.'
Materials and Methods, 1956, vol. 44, Oct., pp. 102–6.

This article contains a practical discussion of the factors which require consideration in using chromium-nickel stainless steels for cold-worked parts. Work-hardening rates of the respective steels of this type are an indication of their relative suitability for cold heading, and in this connexion a chart shows the work-hardening characteristics of typical stainless

steels, as exhibited by their behaviour during wire drawing.

Attention is directed to the advantage of nickel, in stabilizing the austenitic structure and lessening the degree of hardening occurring on cold working: an 18-18 nickel-chromium steel is especially suitable for cold-formed components, particularly where it is necessary to preserve the very low degree of magnetic permeability which is associated with a stably austenitic structure.

The desirability of economy in tool life also underlines the need for a cold-heading type of steel such as the 18-18 composition, which will not work-harden to an extreme degree. Wire for cold heading should be of the lowest possible hardness and should possess a certain amount of stiffness, so that it can be fed smoothly into the cold-heading machine. This condition is produced by annealing the wire to a dead-soft condition, at a size slightly larger than the finished one, coating it with a plating of copper and a lubricant, and cold-drawing to finished size. Care must be taken to ensure a satisfactory finish on the cold-headed parts.

Design of head is also of considerable importance. Attention is called to a detailed study of the influence of this variable on cold heading (*Jnl. Iron and Steel Inst.* 1955, vol. 180, May, p. 51), and a figure in the present article illustrates strain patterns resulting from cold-heading to several basic designs. The degree of local deformation of the cold-worked metal depends upon the shape of the headed part, and it has been found that round heads are more subject to stress cracking than are other shapes. Stress cracks may arise also from poor heading practice, and some recommendations are made on procedure which will obviate such effects and ensure successful production of cold-headed parts.

Nitriding of Stainless Steels

H. A. JOHNSON: 'Nitriding Stainless Steels Costs Less in a Salt Bath.'

Materials and Methods, 1956, vol. 44, Nov., p. 113.

The writer advocates the use of a salt bath containing carbonates and cyanides, as a nitriding medium for case-hardening of various types of steel (including nickel-chromium stainless steels of normal and precipitation-hardening types), and heat-resisting alloys. The material to be treated is suspended in an unagitated bath, and after nitriding the parts are air-quenched and rinsed in water to remove the salt.

Illustrations in the article include graphs showing typical case depths after nitriding under various conditions, and representative hardness gradients for various materials.

Sodium-Hydride Descaling of Nickel-containing Materials

W. F. S. TAYLOR: 'Sodium-Hydride Descaling Process.'

Metal Treatment and Drop Forging, 1956, vol. 23, Nov., pp. 465-8.

The author draws attention to the advantages of the sodium-hydride descaling procedure, discusses the principles on which it operates, and describes the main features of the plant used and of the technique of working.

Typical descaling plant now operating in various works is described and illustrated. The units considered include one used for treating batches of heavily scaled chromium-iron sheets, another which is employed for descaling of stainless-steel wire in coils, and a third which treats sheet and pressings in Nimonic 75 and other heat-resisting alloys. The economic, as well as the technical, aspects of the subject are discussed.

Stress-Corrosion Cracking of Stainless Steels

W. G. RENSHAW: 'Nature of Stress-Corrosion Cracking of Stainless Steels When Other Types of Corrosion are Present.'

Corrosion, 1956, vol. 12, Oct., pp. 477t-8t.

The note shows three photomicrographs illustrating material in which other types of corrosion were associated with stress-corrosion cracking in austenitic chromium-nickel steel. In the first case intergranular attack had occurred in a molybdenum-containing 18-8 steel which had been in contact with various corrosive media, including chlorides and sulphates. Cracks resulting from such exposure had acted as notches, starting stress-corrosion cracking. In the second instance the stress-corrosion cracking was associated with corrosion resulting from exposure to an environment consisting of an organic acid and traces of hydrochloric acid. The third case illustrates pitting corrosion which may occur in carbide-free material in association with stress conditions. When encountered, pitting is always found on the surface which is under tension. If the stresses are mild only pits may occur, but under more severe conditions cracks may be initiated at the base of the pit and spread into the material. A typical case is illustrated.

All three cases examined point to the existence of some factor of a localized nature which precedes actual transgranular stress-corrosion cracking, and probably acts to initiate it. It is not yet known whether some kind of notch effect is essential in all cases of stress corrosion.

Effect of Sulphide Scales on Corrosion of Austenitic Steel

E. L. HILDEBRAND: 'Effect of Sulfide Scales on Catalytic Reforming and Cracking Units.'

Corrosion, 1956, vol. 12, Oct., pp. 73-6.

The fact that high-temperature sulphide attack on steel components of catalytic re-forming equipment differs from sulphur corrosion found in certain other refinery processing units has been well established. The factors to which this difference is due are detailed in this report. Severe losses in carbon and low-alloy steels has been experienced in catalytic re-forming reactors, re-heat furnaces, transfer lines and effluent exchanger equipment.

This report illustrates the effects observed in carbon, chromium-molybdenum and 18-8 chromium-nickel

steel components. The case of the stainless steel concerned a fluid cracking unit in which the steel lines suffered severe intergranular corrosion due to the action of moist sulphide scale during the periods when the plant was not operating. The following recommendations are made with a view to obviating this deterioration:

'Furnace coils should be steamed out in the conventional manner, and steaming should be continued until the 18–8 tubes and lines are filled with a weak alkaline solution. A 3 per cent. sodium-hydroxide solution or a 5 per cent. soda-ash solution is considered suitable. When mechanical work requires draining the tubes, exposure to air should be permitted no longer than is required and the tubes should be re-filled with alkaline solution for pressure testing. It follows that the alkaline material in the tubes should be thoroughly flushed out with fresh water immediately prior to the coming on stream.'

The author is of the opinion that this procedure should minimize the danger of intergranular corrosion, without unduly adding to maintenance work.

Corrosion-Resisting Properties of Stainless Chromium-Nickel-Manganese Steels

W. G. RENSHAW and R. A. LULA: 'The Corrosion Properties of Chromium-Nickel-Manganese Austenitic Stainless Steels.'

Amer. Soc. Testing Materials, Preprint 81, 1956; 24 pp.

This paper contains detailed results of tests in which corrosion-resistance was studied on steels within the following range of composition: chromium 15–21, nickel 2–6·50, manganese 5–17, per cent. The materials were exposed in strongly oxidizing conditions and in environments of moderate severity, and the susceptibility of the steels to localized attack was also investigated. The three main alloy elements were systematically varied in the steels tested, and the corrosion-resisting qualities are evaluated in relation to these individual constituents.

The general conclusions drawn are summarized below:—

'The influence of nickel is prominent in these chromium-nickel-manganese steels. In boiling nitric-acid tests, the improvement due to increasing nickel is more pronounced at 17 per cent. chromium than at a 20 per cent. chromium level. Nitric-acid corrosion rates comparable to those of 18–8 chromium-nickel steel can be attained with the 20 per cent. chromium, 6 per cent. nickel steel containing high manganese, and this composition also shows very good resistance, as does 18–8, to localized attack and to boiling 50 per cent. lactic acid.

Manganese can be varied from 4 to 14 per cent., with no significant effect on corrosion-resistance. On the other hand, boiling-nitric-acid tests and tests in certain organic acids, together with salt-spray results, indicate that increasing chromium produces considerable improvement. In many mild environments, such as acetic, citric and phosphoric acids, Types 201 and 202* chromium-nickel-manganese steel are equal, respectively, to Types 301 and 302 chromium-nickel grades. For more severe conditions, as typified by the boiling-nitric-acid test, or for localized conditions, a 20 per cent. chromium, 6 per cent. nickel, 8 per cent. manganese composition has been developed. This steel is comparable to Type 304 chromium-nickel steel.

'The intergranular corrosion-resistance of the chromium-nickel-manganese steels is the same as that of the chromium-nickel steels. Lowering the carbon contents prevents intergranular corrosion, but only in short sensitizing times: the behaviour in this respect is similar to that of low-carbon Type 304 steel. Carbon content appears, however, to be a less critical factor in the chromium-nickel-manganese than in the straight chromium-nickel grades of comparable compositions.

'Like the chromium-nickel steels, the chromium-nickel-manganese types are subject to stress-corrosion cracking. Resistance to stress-corrosion does not appear to have any association with the amount of chromium, nickel or manganese, provided that the structure is austenitic. The presence of delta ferrite can prevent or lessen susceptibility to this type of failure.'

Corrosion by Sulphuric Acid: Resistance of Nickel Alloys

J. BÜNGER: 'Corrosion by Sulphuric Acid.'

Werkstoffe und Korrosion, 1956, vol. 7, June, pp. 322–30.

In the first part of this paper a wide survey is made of the literature on corrosion by sulphuric acid.

* For compositions of steels referred to by A.I.S.I. numbers, see table below.

Composition of Chromium-Nickel-Manganese and Chromium-Nickel Steels

Designation	C %	Mn %	Cr %	Ni %	N %
AISI 201 ..	0·15 max.	5·50- 7·50	16·0-18·0	3·5- 5·5	0·25 max.
AISI 202 ..	0·15 max.	7·50-10·0	17·0-19·0	4·0- 6·0	0·25 max.
AISI 301 ..	>0·08-0·20	2·00 max.	16·0-18·0	6·0- 8·0	—
AISI 302 ..	>0·08-0·20	2·00 max.	17·0-19·0	8·0-10·0	—
AISI 304 ..	0·08 max.	2·00 max.	18·0-20·0	8·0-11·0	—

The data reported by numerous investigators, on many materials, are correlated and presented in the form of comparable graphs, with critical commentary on the results reported. The survey is supported by reference to original investigations.

This review is followed by a report of tests made by the author, with the two-fold object of determining the influence of the iron content of nickel-molybdenum-base alloys, on their resistance to corrosion by sulphuric acid, and the behaviour, in sulphuric-acid solutions, of alloys and steels intermediate in composition between the nickel-molybdenum-base alloys and the normal 18-8 commercial chromium-nickel steels. In the latter section attention was given to the effects of chromium, nickel, molybdenum and copper.

The corroding solutions covered a wide range of concentration and temperature. The following particulars are given of the materials for which detailed test data are shown: the compositions were representative of alloys made by the firm with which the author is associated (Edelstahlwerke Schmidt und Clemens, Berghausen).

Designation	C %	Cr %	Ni %	Mo %	Si %	Cu %	Fe %
Märker S.N.18 ..	0·08	18	18	2	—	2	bal.
„ S.N.25 ..	0·10	18	25	4	—	2	bal.
„ S.N.42 ..	—	18	42	5	—	2	bal.
Euzonit 50	—	—	55	20	—	—	20
„ 60	0·10	17	60	20	—	—	bal.
„ 70	—	—	67	30	—	—	bal.
„ 85	—	—	85	—	9	4	—

The results are summarized in a graph comparing the behaviour of the respective materials on the basis of a corrosion rate of 0·5 mm./yr. in sulphuric acid of various concentrations. The curves demonstrate the particular suitability of the 67-30 nickel-molybdenum alloy for use in contact with a wide range of solutions.

Fatigue of Stainless Steel in Contact with Liquid Sodium

J. W. MARTIN and G. C. SMITH: 'A Preliminary Study of the Fatigue of Metals in Liquid Metal Environments.'

Metallurgia, 1956, vol. 54, Nov., pp. 227-32, 238.

The likelihood of the increasing use of liquid-metal coolants in power plants of various types emphasizes the importance of establishing information on the fatigue properties of metals and alloys in contact with such media. This paper records tests of the fatigue-resistance, at room temperature, of amalgamated copper alloys (70-30 and 60-40 brass, copper-tin and copper-cadmium), and of the behaviour, at 300°C., of mild steel in contact with liquid tin and 18-8 chromium-nickel steel in contact with liquid sodium. In the elevated-temperature tests the corrodent metal was brought into contact with the steels by sealing it into a 0·25-in. diameter hole bored along the axis of the test specimens. Methods for insertion of the metals and for sealing are described.

It was found that:

In brass specimens amalgamation of the surface lowered fatigue life, but with the copper-tin and copper-cadmium alloys there was little, if any, effect;

Contact with liquid tin shortened the fatigue of life of mild steel;

Although there was considerable scatter in the results on the stainless-steel samples, the general indication was that the S-N curve for the sodium-filled specimens was closely similar to that for the unfilled samples. There was, however, some cracking in the sodium-filled test pieces.

Reactions of Molten Sodium Hydroxide with Nickel and Nickel-containing Alloys

See abstract on p. 2.

Corrosion by Fertilizers

D. C. VREELAND and S. H. KALIN: 'Corrosion of Metals by Liquid Fertilizer Solutions.'

Corrosion, 1956, vol. 12, Nov., pp. 569t-75t.

The tests reported were made to establish the suitability of various materials for use in contact with 'nitrogen fertilizer solutions' (which consist of aqueous solutions of ammonia, ammonium nitrate or urea, or combinations of the three, as sources of nitrogen), and 'complete-mix liquid fertilizers' (which may contain various amounts of ammonia, phosphoric acid, potassium chloride, urea and ammonium nitrate).

The materials tested included plain carbon steels, straight-chromium steels, chromium-nickel steels of 18-8, 18-8 low-carbon and 18-8 molybdenum-containing types, and aluminium alloys. All were representative of commercially available materials. Full analyses are given, and the compositions of the fertilizer solutions are also tabulated. The tests were of about one month's duration, and the solutions were used at 100°-150°F. (38°-65°C.).

Most of the materials were evaluated by two types of corrosion test:—

(1) Partial-immersion tests of small specimens in flasks containing the fertilizers.

(2) Simulated-service tests, in which small tanks of each material to be tested were fabricated by welding and partially filled with the fertilizer solution.

The results of the two types of corrosion test are individually reported and discussed, with tables and graphs showing rate of corrosion, maximum penetration, and the nature of the attack undergone.

The findings indicate that 12 per cent. chromium steel, all the chromium-nickel stainless steels, and aluminium, should be suitable as constructional materials for tanks for nitrogen liquid fertilizers, and that the chromium-nickel stainless steels can be used for tanks holding complete-mix solutions. It is recommended, however, that service tests should be conducted before any individual material is adopted for such application.

Special Corrosion-Resisting Applications of Nickel and Nickel Alloys

H. O. TEEPLE and R. M. FULLER: 'New Chemical Products and High-Nickel Alloys.'

Industrial and Engineering Chemistry, 1956, vol. 48, Nov., pp. 1971–4.

The article deals with the uses of nickel and nickel alloys in plants for manufacture of chemical products, with special reference to their use in some of the more recently introduced processes. The materials considered in the review are nickel of various grades, nickel-copper alloys of the Monel types, nickel-chromium-base alloys of the Inconel group, nickel-molybdenum alloys (Hastelloy B and Chlorimet 2), nickel - chromium - molybdenum - tungsten alloys (Hastelloy C and Chlorimet 3), and nickel-silicon-base alloys of the Hastelloy D type.

The nominal compositions of the respective materials are given, together with notes on their general characteristics. Applications for which one or more of these materials are specially suitable are then reviewed, with particular reference to their employment in applications involving contact with chlorine, iodine, fluorine, hydrogen fluoride, hydrofluoric acid, bromine, alkalis, pharmaceutical products, synthetic detergents, and acids.

A selected *bibliography* supports the text.

Welding of Stainless-Steel Piping

E. B. LAVELLE, L. H. RASMUSSEN and E. M. KUCHERA: 'Root Passes in Stainless Steel with New Inert-Arc Procedures.'

Welding Jnl., 1956, vol. 35, July, pp. 647–54.

The authors briefly review the shortcomings of methods in current use for joining piping, and describe a new procedure developed for welding pipe and tubing which must meet the stringent requirements imposed for components of atomic-energy plant.

Welding of Titanium to Stainless Steel

F. W. McBEE, J. HENSON and L. R. BENSON: 'Problems Involved in Spot Welding Titanium to Other Metals.'

Welding Jnl., 1956, vol. 35, Oct., pp. 481S–7S.

In view of the increasing importance of titanium in various fields of engineering, considerable interest is developing in the welding of this metal to other materials. The information presented in this paper consists of a collection of data on the properties of welds between titanium and stainless steels or other metals: it also contains a discussion of some of the problems involved in joining titanium to dissimilar metals.

The ultimate objective of the research described was the successful spot welding of titanium to stainless steel. Preliminary investigation had indicated that in most cases such welds showed very poor properties and work was therefore planned in three sections:—

The first phase of the study dealt entirely with the properties of welds between titanium and 18–8 stainless steel.

The second phase was concerned with welds between titanium and the individual metals contained in stainless steel. Each component (iron, chromium, nickel) was independently investigated, to determine whether one particular element in this group might be responsible for the troubles encountered in obtaining satisfactory welds.

The third phase of the research covered welding of titanium to zirconium, aluminium or magnesium.

Throughout the investigation the materials used were varied in physical properties, as well as in the characteristics which are of significance in relation to resistance welding. Selection of the metals welded was based on their melting point, thermal conductivity, electrical resistivity, crystalline structure, location in the periodic table, and atomic diameter. These properties of the respective materials are recorded in the introduction to the report.

Full details are given of the results of each series of experiments, on the basis of which the following conclusions are drawn:—

'1. Attempts to secure a better heat balance by means of a high-resistance electrode produced little or no improvement in the characteristics of titanium/stainless-steel welds.

'2. The investigations on the individual components of stainless steel suggest that the incompatibility of the titanium/stainless steel weld is probably due to the iron content of the steel. Of the three main components of stainless steel, chromium produced a far better weld with titanium than was obtained with either iron or nickel.'

(The authors are at present unable to suggest any theory to explain this difference.)

'3. Due to the somewhat similar properties of titanium and zirconium, welds between these two materials gave the highest strength of any combinations tested. The ductility, however, remained low.

'4. Magnesium or aluminium, when spot-welded to titanium, produced braze-type joints having fair characteristics.

'5. The injection of additional chromium into the titanium/stainless-steel weld had little effect on the characteristics of the joint.'

Welding of Dissimilar Materials

See abstract on p. 1.

Nickel-Alloy Brazing Filler Materials

W. H. CHANG: 'Basic Characteristics of Some Heat-Resisting Brazing Filler Materials.'
Welding Jnl., 1956, vol. 35, Sept., pp. 431S–43S.

This paper summarizes the results obtained in a preliminary evaluation of four filler materials potentially useful in brazing for high-temperature applications. The evaluation was based on room-temperature tensile properties of butt-brazed joints.

The compositions of the four commercial filler materials examined, together with other relevant details, are given in Table I.

Consideration of all the requirements of the investigation led to the selection of a rectangular butt-type specimen $\frac{1}{2} \times 2 \times 3$ in. overall dimensions: two standard 0·250-in. tensile bars and one metallographic section could be prepared from each brazed joint. Particular attention was given to the method of applying the filler material: the procedure used is described and illustrated. Preliminary studies on the influence of surface preparation, on the properties of the joints, indicated that grinding of the specimens was preferable to blasting. For the main tests, therefore, grinding was adopted.

The suitability of the respective filler materials was investigated as a function of brazing time, brazing temperature and atmosphere, intergranular penetration of the base material, and, in particular, the effect of joint clearance on room-temperature tensile properties. The results of the tests, which are reported in detail and well illustrated, led to the following conclusions:—

Table I

Filler Material	Nominal Composition %	Approximate Liquidus °F.	Approximate Liquidus °C.	Form in which applied
Ni-Cr-B Alloy No. 1 ..	Cr 15, Si 4, B 3·5, C 1·0, Fe 4·0, Ni bal.	1925	1050	−325 mesh powder
Ni-Cr-B Alloy No. 2 ..	Cr 7, Si 4·5, B 3, Fe 3, Ni bal.	1825	995	−250 mesh powder
Ni-Cr-Si Alloy	Cr 19, Si 10, Fe 5·0, Ni bal. ..	2035	1112	−200 mesh powder
Au-Ni Alloy	Au 82, Ni 18	1742	950	$\frac{1}{32}$-in. wire

The choice of these materials as representative was based on the characteristics of the respective alloys which affect their use in brazing: these characteristics are discussed as a preliminary to the report of the experimental work.

The base materials used were of three types, ranging from a straight-chromium steel to a complex nickel-base alloy, see Table II:—

Table II

Base Material	Nominal Composition %
N-155	C 0·15, Ni 20, Cr 20, Co 20, Mo 3, W 2·5, Nb+Ta 1, Al 0·15, Fe bal.
AISI 347	C 0·08, Si 0·75, Mn 2·0, Ni 10, Cr 18, Nb + Ta 0·8, Fe bal.
AISI 405	C 0·09, Si 0·5, Mn 0·5, Cr 12, Ni 0·5, Al 0·2, Fe bal.

Brazing was carried out in an atmosphere of purified hydrogen, in a box placed inside the furnace. No flux was used. As measured at the entrance to the brazing box, the hydrogen had a dew point below −80°F. (−62°C.). Brazing conditions are described in detail.

At 'zero' clearance butt joints in all three materials brazed with the nickel-chromium-boron and nickel-carbon-silicon filler materials showed satisfactory tensile properties. In the chromium-steel specimen failure was in the base material and in the other two alloys it occurred at the joint. The austenitic-steel joints showed a tensile strength of 85,000 p.s.i. (38 tons per sq. in.) and elongation of 20–40 per cent.: the joints in the high-nickel alloy had tensile strengths of 95,000–100,000 p.s.i. (42·4–44·6 tons per sq. in.) with elongations of 17–35 per cent. The joints brazed with the nickel-chromium-silicon alloy were slightly inferior to those made with the nickel-chromium-boron alloys.

The joints referred to above suffered severe embrittlement at clearances of 0·002 in. or over: elongation fell to 0–5 per cent. and the tensile strengths were only 30–70 per cent. of those obtained at 'zero' clearance. This embrittling effect was more pronounced with the nickel-chromium-silicon alloy than with the nickel-chromium-boron type.

The tensile properties of butt joints brazed with the gold-nickel alloy showed, however, no sensitivity to clearance. From 'zero' to 0·006-in. clearance, both the chromium steel and austenitic steel specimens failed in the base materials, and the nickel-alloy specimens, although fractured at the joint, exhibited, throughout the clearance range, tensile properties

equal to those of the base material exposed to the corresponding brazing cycles. The average elongation was 25 per cent.

The deleterious effect of clearance on joint properties which was observed with the nickel-chromium-base filler materials was found to originate from the presence of brittle phases in the joints, resulting from the complex constitution of such brazing alloys. The embrittlement is aggravated by the tendency of these phases, particularly those associated with the nickel-chromium-silicon alloy, to solidify into a continuous layer. At 'zero' clearance occurrence of the brittle phases is prevented by diffusion, into the base material, of the alloying elements which would be responsible for forming such phases.

Irrespective of clearance, joints made with the gold-nickel alloy showed similar microstructures, consisting essentially of a mixture of gold-nickel solid solutions.

The boron-containing filler materials caused intergranular base-material penetration: the more complex the base material the more extensive is such penetration, but there are indications that this type of penetration does not occur in base materials which do not contain elements forming stable borides, e.g., chromium, molybdenum, tungsten and cobalt.

Welding of Clad Steels: Recommended Procedure

J. HINDE: 'The Welding of Clad Steels.'

Mond Nickel Co., Ltd., Publn. 1032A, 1956; 19 pp.

Based on article published in *Welding and Metal Fabrication*, January and February 1956: see abstract in *Nickel Bulletin*, 1956, vol. 29, No. 7, p. 150.

PATENTS

Irradiation of Materials during Precipitation Hardening

The invention described is based on the observation that in precipitation hardening it is possible to use lower temperatures of treatment if the materials are subjected, during such treatment, to neutron irradiation or bombardment in an atomic pile. Specific reference is made to two high-alloy nickel-chromium steels treated by this method. Such steels can be precipitation-hardened at 300°–400°C. if subjected to such conditions, whereas otherwise a minimum temperature of 600°C. would be required.

W. JESSOP AND SONS, LTD. (inventor, G. T. HARRIS).
Brit. Pat. 762,174.

Sintered Plates for Alkaline Accumulators

Sintered plates are prepared by depositing a suspension of fibrous metallic powder (preferably nickel or iron-nickel alloy produced by thermal decomposition of the carbonyls) in an aqueous solution of the sodium salt of carboxymethylcellulose, onto a perforated metallic band, preferably wire mesh of specified dimensions. The coated mesh is passed between two scrapers adjusted to permit production of the required thickness, dried, and cut to plates of the desired size.

STÉ. DES ACCUMULATEURS FIXES ET DE TRACTION.
Brit. Pat. 759,788 (similar to *French Pat.* 1,077,036).

The negative electrode for alkaline accumulators consists of a porous sintered skeleton of a nickel-copper alloy containing up to 50 per cent. of nickel. The electrode is formed from intimately mixed powders of the two metals and it is specified that a high proportion of the particles of the powder shall be of controlled grain size. The powder mixture is preferably produced by decomposition of nickel-carbonyl vapour in the presence of copper powder.

ACCUMULATOREN-FABRIK A.G. *Brit. Pat.* 765,073.

Deposition of Metals from the Carbonyls

The rate of deposition of metals from their carbonyls is accelerated by the inclusion, in the vapour phase, of silicon tetrachloride. As a preferred example of the process, the deposition of nickel from the carbonyl vapour in the presence of silicon tetrachloride is described, for deposition of nickel on a polystyrene base.

M. V. SULLIVAN, assignor to BELL TELEPHONE LABORATORIES, INC. *U.S. Pat.* 2,759,848.

Production of Nickel Foil by Thermal Decomposition of the Carbonyl

The surface of an endless metal belt, e.g., of aluminium or copper, is oxidized, and subsequently brought into contact with gaseous nickel carbonyl, preferably mixed with CO_2. The temperature of the belt is then raised to 300°–400°F. (150°–205°C.) causing decomposition of the carbonyl and deposition of metallic nickel on the belt. The thin nickel film so produced can be stripped off as a continuous sheet of metal.

A. H. CLARKSON (communication from COMMONWEALTH ENGINEERING CO. of OHIO).
Brit. Pat. 763,411 (similar to *U.S. Pat.* 2,701,901).

Bright Nickel-Plating Solutions of High Levelling Capacity

Solutions claimed to give high levelling and good brightness in the deposit contain a nickel salt, sulphonated phthalide and/or sulphonated benziso-benzofuranone and a salt of at least one of the metals zinc, cadmium or thallium. Operating conditions are pH 3·1, temperature 135°F. (57°C.), cathode current density 50, anode current density 20 amp./sq. ft.

ROCKWELL SPRING AND AXLE CO.
Brit. Pat. 763,981 (similar to *French Pat.* 1,104,411).

Titanium-Clad Steel

In the cladding of steel with titanium an electro-deposited interlay of iron or nickel is used.

LUKENS STEEL CO. *Brit. Pat.* 759,413.

See also—

Brit. Pat. 759,468, which claims the product made according to *Brit. Pat.* 759,413, and 759,469, which covers the pack used in the process.

Electroless Deposition of Nickel

Non-metallic bodies are prepared for electroless plating by roughening the surface and coating this surface with catalytic material in small particles. Such coating may be deposited by contacting the basis material with a solution of a salt of the catalytic metal and subsequent reduction to the metallic condition. The catalytic particles may be of carbon, silver, iron, cobalt, nickel, aluminium, copper, gold, platinum or palladium. The treated base is then plated in an aqueous solution of a nickel salt and a hypophosphite.

GENERAL AMERICAN TRANSPORTATION CORPN. (inventors, G. GUTZEIT, W. J. CREHAN and A. KRIEG). *Brit. Pat.* 761,556 (similar to *U.S. Pats.* 2,691,401 and 2,691,403; *Belgian Pat.* 522,181; *French Pat.* 1,088,263; *South African Pat.* 18,898; *Australian Pat.* 165,650).

Regeneration of Electroless Nickel-Plating Solutions

Solutions containing nickel ions and hypophosphite ions are regenerated by adding nickel hypophosphite and $H(H_2PO_2)$ or alkaline hypophosphites, and a soluble alkali, to minimize the introduction of extraneous ions and the build-up of undesirable salts.

GENERAL AMERICAN TRANSPORTATION CORPN. *U.S. Pat.* 2,766,138.

Heat-Treatment of Nickel-Copper-Zinc Alloys

British Patent 706,502 described a copper-base alloy containing nickel and zinc with small amounts of cadmium, and with or without some other elements. This later patent provides for widening the composition and for hardening treatment, which comprises heating at 50°-500°C., preferably after rolling. Typical compositions are given.

W. M. WALLER and P. H. WALLER. *Brit. Pat.* 761,561 (addition to 706,502).

Copper Alloys for Springs

An alloy claimed to be particularly suitable for current-carrying springs, including spring-contact members such as are used in electrical computing equipment, contains nickel >5-15, silicon 0·1-2, aluminium 0·1-6, and/or magnesium 0·1-2, per cent., copper balance. Such alloys are hardened by precipitation of nickel silicide, as resulting from ageing at 750°-1000°F. (400°-540°C.).

For the purpose mentioned, such alloys are considered to be preferable to beryllium-copper: some comparison is made between the properties of the two types of material.

INTERNATIONAL BUSINESS MACHINES CORPN. *Brit. Pat.* 761,110.

Processing of Magnetostrictive Alloys for Magnetic Cores

The patent refers to treatment designed to enhance the properties of magnetostrictive alloys containing nickel 40-60, silicon 0-4, manganese 0-2, molybdenum 0-4, cobalt 0-5, per cent., balance iron. After melting in air, gas or vacuum, or sintering, the alloy material is hot-rolled to give a product less than 10 mm. thick, and subsequently cold-rolled, to effect, with the hot-rolling, a total reduction of 97 per cent. The result is a strip not more than 0·20 mm. (preferably not more than 0·15 mm.) thick. In addition, insulation of the laminae is covered, also subsequent annealing in pure dry hydrogen and the use of the cores in apparatus which utilizes the magnetostriction phenomenon in detection or recording of mechanical effects.

STÉ. MÉTALLURGIQUE D'IMPHY. *Brit. Pat.* 761,731.

Dense ('Heavy') Alloys containing Nickel

Dense alloys suitable for balance weights and other parts in which high weight/mass ratio is essential consist of tungsten and/or molybdenum not less than 75 per cent., together with iron, and nickel and/or cobalt. Nickel + cobalt : iron = 3 : 2-1. Chromium up to 15 per cent. may also be present. The preferred composition is given as tungsten 90, nickel 6-7·5, iron 2·5-4, per cent. Powder-metallurgical methods of production of the alloy are also claimed.

GENERAL ELECTRIC CO., LTD. (inventors, E. C. GREEN, D. J. JONES and W. R. PITKIN). *Brit. Pat.* 760,113.

Welding of 90-10 Copper-Nickel Alloys

In inert-gas-shielded arc-welding of iron-containing 90-10 copper-nickel alloys, sound, strong, ductile welds can be made by using a rod containing nickel 5-15, iron 0·1-2, manganese 0·1-1, silicon 0·05-0·5, titanium 0·02-0·5, per cent., copper balance.

In this connexion see *Nickel Bulletin*, 1956, vol. 29, Nos. 10-11, p. 185.

G. R. PEASE and T. E. KIHLGREN, assignors to INTERNATIONAL NICKEL CO., INC. *U.S. Pat.* 2,772,963.

Copper-Nickel Brazing Alloys containing Lithium and Boron

Self-fluxing brazing alloys, which can be operated in air, contain lithium 0·25-8, boron 0·25-2·5, nickel 5-50, per cent., copper balance. In addition to their use in joining steels and other metals, such alloys may be used for brazing ceramics to metals, and are suitable for deposition of metal coatings by spray techniques and for use in dip coating of various steels.

N. BREDZS, assignor to LITHIUM CORPN. OF AMERICA, INC. *U.S. Pat.* 2,768,893.

Nickel-Manganese Brazing Alloys

Nickel-manganese alloys have melting points which render them suitable for brazing stainless steel or nickel-chromium-(cobalt) alloys, and the wetting and flow characteristics of the alloys are also satisfactory. The creep strength of brazed joints made with such materials is improved by adding, to the binary manganese-nickel alloy, palladium and/or molybdenum and/or tantalum. A preferred composition is given as manganese-nickel 40 : 60, with tantalum 10 per cent. (see also *Brit. Pat.* 763,172).

BRISTOL AEROPLANE CO., LTD. (inventors, G. LLEWELYN and R. G. UBANK). *Brit. Pat.* 763, 171.

Hard Surfacing of Titanium

A hard surface layer is formed on titanium by applying a thin layer of powdered nickel, manganese or chromium or of their alloys (preferably nickel), in a liquid organic vehicle of a volatile nature, and heating the material so treated to a temperature below the melting point of the titanium and of the powder, to cause diffusion.

The faced material is then quenched and is tempered at 800°F. (425°C.). A typical example relates to the nickel coating of titanium and subsequent lacquering, in which case the resulting facing is stated to have a maximum hardness of 620 D.P.H.

GENERAL MOTORS CORPN., assignee of C. W. VIGOR. *Brit. Pat.* 763,909.

Anisotropic Permanent-Magnet Alloys

In iron-nickel-aluminium-cobalt magnet alloys it has previously been found that the highest degree of anisotropy is obtained only with high cobalt contents, e.g., 22–25 per cent. Such high cobalt inevitably renders the alloys expensive. British Patent 750,015 specified anisotropic permanent-magnet alloys in which high magnetic anisotropy is obtained with relatively low cobalt (see *Nickel Bulletin*, 1956, vol. 29, No. 7, p. 152). The present claim covers the manufacture of magnets having the advantages covered in 750,015, together with somewhat higher coercive force. According to these claims, the magnets are made from an iron-base alloy containing aluminium 5–11, nickel at least 10 but less than 22, cobalt 10–25, copper up to 7, niobium and/or tantalum 0·2–7·0, silicon 0·2–3, per cent., balance iron. It is specified also that the aluminium content shall be less than that of the nickel, and there is a definite ratio of silicon to cobalt. The alloys may contain small amounts (less than the total of 2 and not more than 1 per cent. of each) of carbon, molybdenum, silver, sulphur, phosphorus, and may further contain one or more of the elements tungsten, manganese, chromium, titanium and zirconium.

PHILIPS ELECTRICAL INDUSTRIES, LTD. (inventors, W. T. DEAN and A. STUTTARD).
Brit. Pat. 763,142 (Addition to 750,015).

Glass- (or Ceramic-) to-Metal Joints

Joints between ceramic bodies and metal, or between a specified type of borosilicate glass and nickel-cobalt-iron alloys of the Nilo K type are produced by coating the dielectric material with a nickel-chromium alloy and brazing the metal to the coating. The alloy, which may be of the 80–20 nickel-chromium type, is deposited by thermal evaporation in vacuo.

METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (inventor, F. ASHWORTH). *Brit. Pat.* 761,662.

High-Tensile Steels for Welding

The basis of the invention is the use of vanadium in place of the molybdenum usually present in low-alloy high-tensile steels. The limits of composition given are carbon 0·13–0·17, silicon up to 0·3, manganese 0·65–0·95, sulphur 0–0·035, phosphorus 0–0·035, nickel 0·5–0·7, chromium 0·8–1, vanadium 0·1–0·25, per cent., iron balance.

BIRMINGHAM SMALL ARMS CO., LTD. (inventors, D. A. OLIVER and D. HARDWICK). *Brit. Pat.* 760,946.

High-Temperature Alloys of Relatively Low 'Strategic-Metal' Content

The following composition limits are specified for high-strength wrought alloys for use in gas-turbine components and other high-temperature applications:—cobalt 18–22, nickel 26–30, chromium 18–22, tungsten 6–7·5, molybdenum 1·25–2·25, titanium 3–4, carbon 0·15–0·25, iron 18–22, per cent. Stress-rupture and fatigue properties at 815°C. are quoted.

J. D. NISBET, assignor to GENERAL ELECTRIC CO. *U.S. Pat.* 2,765, 226.

Boron Additions to Stainless Steels

Improved hot-working properties are obtained in stainless steels by adding boron, in the form of pre-alloys or as boric oxides or borates. The residual boron content is preferably 0·00005–0·004 per cent. The process is directed especially to the treatment of steels of high-nickel-chromium content containing, in addition, smaller amounts of manganese, silicon, copper, cobalt and other elements.

ARMCO INTERNATIONAL CORPN. (inventor, D. L. LOVELESS). *Brit. Pat.* 760,926.

Composition of Stainless Steel for Use in Powder Metallurgy

In production of stainless-steel parts, e.g., filters, by powder metallurgy, the powder used contains 0·5–1·25 per cent. of carbon, to broaden the solidus-liquidus range of the metal, in order to promote correct sintering under conditions which can be adequately controlled in commercial operation. The carbon is subsequently removed by decarburization, giving, in the finished article, properties which are unaffected by the initial presence of the carbon.

A typical composition of the powder is given as chromium 18, nickel 11, molybdenum 2·5, manganese 0·25, silicon 5, carbon 0·6, iron 62–65, per cent.

FEDERAL-MOGUL CORPN. *Brit. Pat.* 762,267.

Nickel-Silicon-base Alloys for Chemical Equipment

Alloys specially resistant to sulphuric acid, and having improved resistance to failure by mechanical or thermal shock, are of the following composition:—silicon 4–10, tantalum 2–25, copper 0–4, iron 0–15, per cent., nickel balance. Aluminium up to 3 per cent. may also be present, provided that the tantalum+iron content is at least 5 per cent. If the tantalum+niobium does not exceed 25 per cent. niobium may replace up to half of the tantalum. Typical corrosion-resisting and mechanical properties are tabulated for various alloys within the range specified.

ELECTRIC FURNACE PRODUCTS CO., LTD., assignee of W. O. BINDER and C. R. BISHOP. *Brit. Pat.* 762,545.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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